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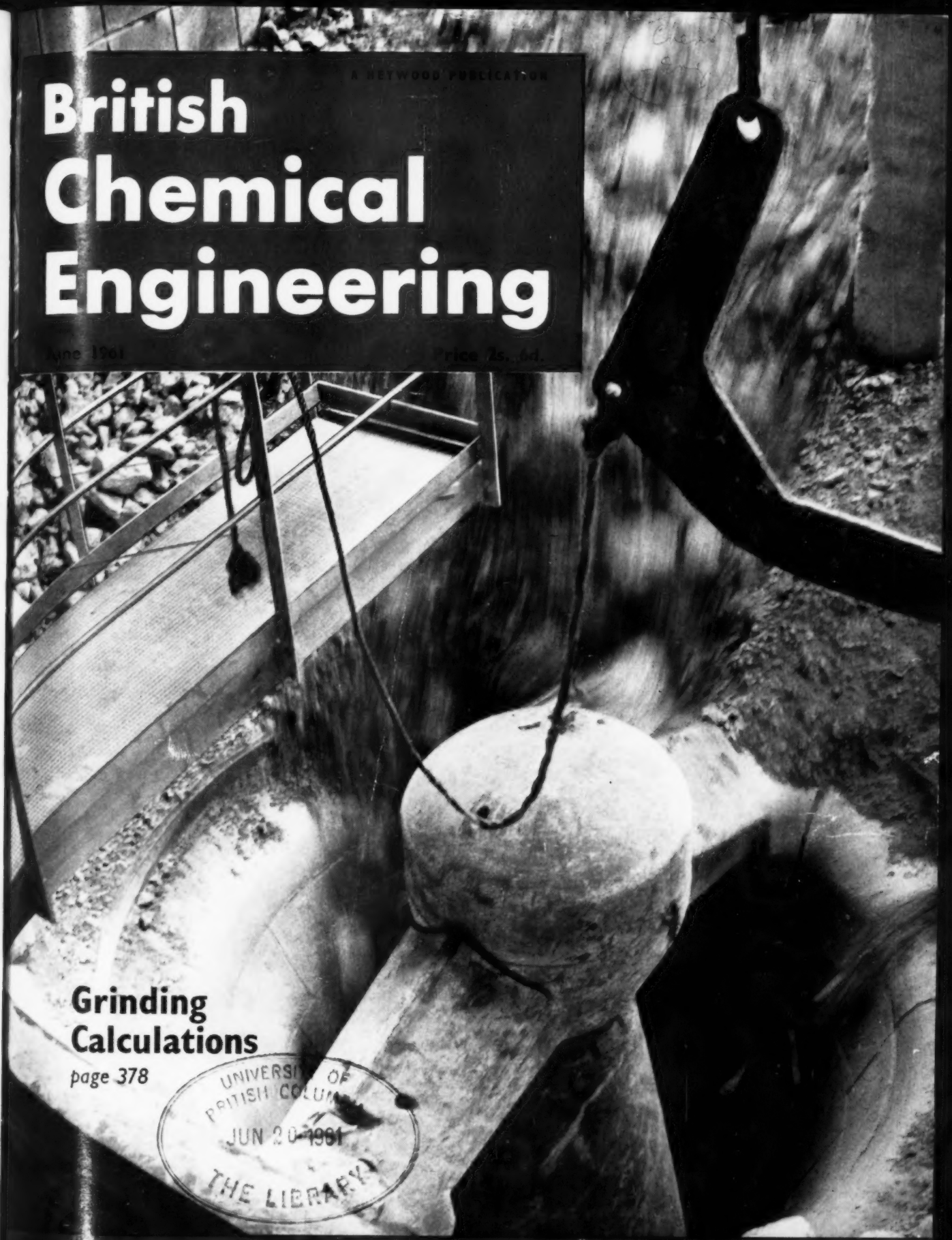

Grinding Calculations

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Sprays Formed by Flashing Liquid Jets

UNTIL recently there has been little data available on the physical process of disintegration that occurs when a spray is formed by flash evaporation from a high pressure zone to a low pressure zone.

By studying the mechanism of sprays formed by the flashing of cylindrical jets, and the sprays themselves, RALPH BROWN and J. LOUIS YORK* have been able to cast much light on the subject.

Their experimental work was carried out on plain cylindrical jets, some with a sharp edged orifice giving a smooth-surface jet with cold water, and some with a drilled hole giving a rough-surfaced but stable jet with cold water. Flashing to any marked degree did not occur unless a temperature substantially above the saturation temperature was provided. With the rough nozzle, disintegration began at the nozzle discharge, whereas with the sharp-edged orifices a delayed action disintegration was observed which occurred several diameters downstream. This is probably explained by the formation of low-pressure eddies at the rough nozzles, which serve as low-pressure stagnation points which may nucleate bubbles.

The experiments revealed that a jet of large diameter may shatter at a lower supherheat than a jet of smaller diameter, a result consistent with existing theory on the stability of liquid jets.

The characteristics of the sprays, too, were investigated and in general it was found that the sprays produced by flashing water are roughly similar to those produced by swirl-chamber nozzles; however, the mean drop sizes were larger (at a distance 6 in downstream from the nozzle) than for gas-atomised sprays, and somewhat smaller than sprays produced by most types of spinning-disc atomizer.

The mean drop sizes are shown to decrease slightly with increasing Weber number at given temperature and therefore at a constant bubble growth-rate constant. At constant Weber number, there is a slight decrease in drop size with increasing temperature, and across the spray, the size tends to increase with distance from the spray axis.

* University of Michigan. Paper presented to 44th A.I.Ch.E. Meeting.

Chemical Reactions and Optical Masers

IT is well known that many, in fact most, reactions proceed faster at high temperatures than at low temperatures. Thus, if a gas containing two constituents that can react is heated, the reaction will occur more quickly. Unless the reaction produces so much heat that it runs away, the speed of reaction can be controlled by the external temperature.

The temperature is just a measure of the degree of motion of the molecules. In particular, it is a measure of the amount of internal vibration of the molecules. This vibrational energy can be increased by absorbing light whose frequency is the same as the characteristic molecular vibration. By absorbing enough of such resonant light, the molecular vibration would be greatly increased, thus making the molecules effectively hotter. This is a very selective process, because every molecule, and even every isotopic species of the molecule, has its own characteristic frequencies and can absorb no other. Thus, under the right conditions, it will be possible to heat up one isotopic species without exciting other isotopic species of the same molecule.

A lot of light of just the right wavelength for the particular molecule would be needed. The optical maser may make this possible, since optical masers are enormously brighter than any other light sources. There is, however, the additional stringent requirement for exactly the right wavelength. At present, only eleven wavelengths have been generated by optical masers, and there has not yet been any attempt on the part of those mainly responsible for the development at, Bell Laboratories, to match them to any molecular vibration.

Mechanical Seals for Abrasive Liquids

THE handling of abrasive fluids in centrifugal pumps is a perennial problem requiring for its solution not only adequate mechanical design of the pump, but also the use of effective mechanical seals. According to R. D. NORTON of Dean Bros. Pumps Inc. Indianapolis, speaking at the recent International Conference on Fluid Sealing*, manufacturers in the United States have not kept pace, in the design of centrifugal pumps, with the heavier unbalanced loads that have resulted from changes in chemical processing during the last 20 years: changes such as the changeover from batch to continuous processing, involving smaller flows and larger unbalanced radial loads; replacement of compression packing by mechanical seals which provide no support against radial load; and automatic regulating valves involving higher differential pressures.

As regards the seals themselves, there are three basic types: double seal, single inside seal and single outside seal. Single inside seals can usually withstand higher pressure than the outside type, but the latter have the advantage that the springs are not exposed to the abrasive environment, and moreover they can be serviced without dismantling the pump. The double seal is generally used where only a negligible amount of diluent can be permitted to enter the pumpage. Although it covers a wider pressure range and may be more effective on high temperature abrasive liquids than a single seal, the double seal has various design and installation disadvantages, and costs more. For these reasons the use of double seals on abrasive liquids is decreasing.

For satisfactory performance of single seals one has to combat the action of solids formed by crystallization, by

scaling, or by settling of solids carried over in suspension. Crystals forming between the faces on shutdown can be counteracted by lubricating the faces; crystallization during running can be reduced on the inside type seal by using a buffer zone liquid or, on the outside seal, by a steam condensate drip or a water smother across the seal faces. Scaling effects can often be counteracted by lowering the temperature by water cooling or by metallurgical methods. Slurries are usually most effectively dealt with by providing a clean seal environment.

Clean Working Conditions Essential

Three courses are open for maintaining a clean seal environment: one may (a) use a clean external flush liquid, (b) filter and recirculate the abrasive pumpage to the seal area, and (c) in the case of high melting point liquids, apply adequate heat to the liquid in the seal area.

As regards flushing, in chemical process work it is rarely possible to permit unlimited flow of flushing liquid and an alternative has to be found. One method is to use a lip seal, generally PTFE, at the throat of the stuffing box, so that the clean flush liquid flows past the lip to the impeller. When the pump shuts down the lip excludes abrasive particles from entering the seal cavity.

Where a clean external flush liquid cannot be introduced to the seal cavity, a filtering recirculation system can be used. This is most useful when solids contamination is infrequent or very small in quantity — a steady flow of solids would require a very large filter area.

* British Hydromechanics Research Association, International Conference on Fluid Sealing, April 1961, Paper H3.

Space Air for Cosmonauts

IT seems appropriate in view of man's entry into the era of space travel and also in view of the forthcoming first British symposium on biochemical engineering to focus attention upon the problem of providing a life-supporting atmosphere for the first cosmonauts.

One process at which a number of interested parties are taking a close look as offering a possible solution to this problem, is the 64 year old Sabatier process, intended originally for the production of methane, but now of interest as a source of water. The point about this is that water can yield oxygen, the latter being harder to come by on a space vehicle than the former. Boeing, for example, are using the Sabatier process in a reactor that simulates the transformation of exhaled carbon dioxide, mixed with hydrogen from an electrolytic cell. It is thought that the reactor and electrolytic cell can form part of a proposed chemical oxygen recovery system to be integrated into a larger, closed-loop life supporting system. In such a system, food, water and air would be used, re-converted and re-used again and again.

A number of closed or partially regenerative systems for space travel are under study, for instance, a safe efficient method of re-using human waste to grow food, provide oxygen from algae plant life and furnish pure drinking water has already been developed. Behind all the research is the need to eliminate stored supplies, in view of the great power required to lift matter into space.

In theory, the reactor of the Boeing oxygen recovery system uses hydrogen from the electrolytic cell and carbon dioxide from the cosmonaut's exhaled breath to produce water and methane. The electrolytic cell, through the electrolysis of water, produces hydrogen and oxygen. By channelling the oxygen to be mixed with the space man's air supply and sending the hydrogen to the reactor, along with the carbon dioxide from more expended air, a full circle has been achieved, apart from utilising the methane. Methods of using the latter in some way as fuel are being studied.

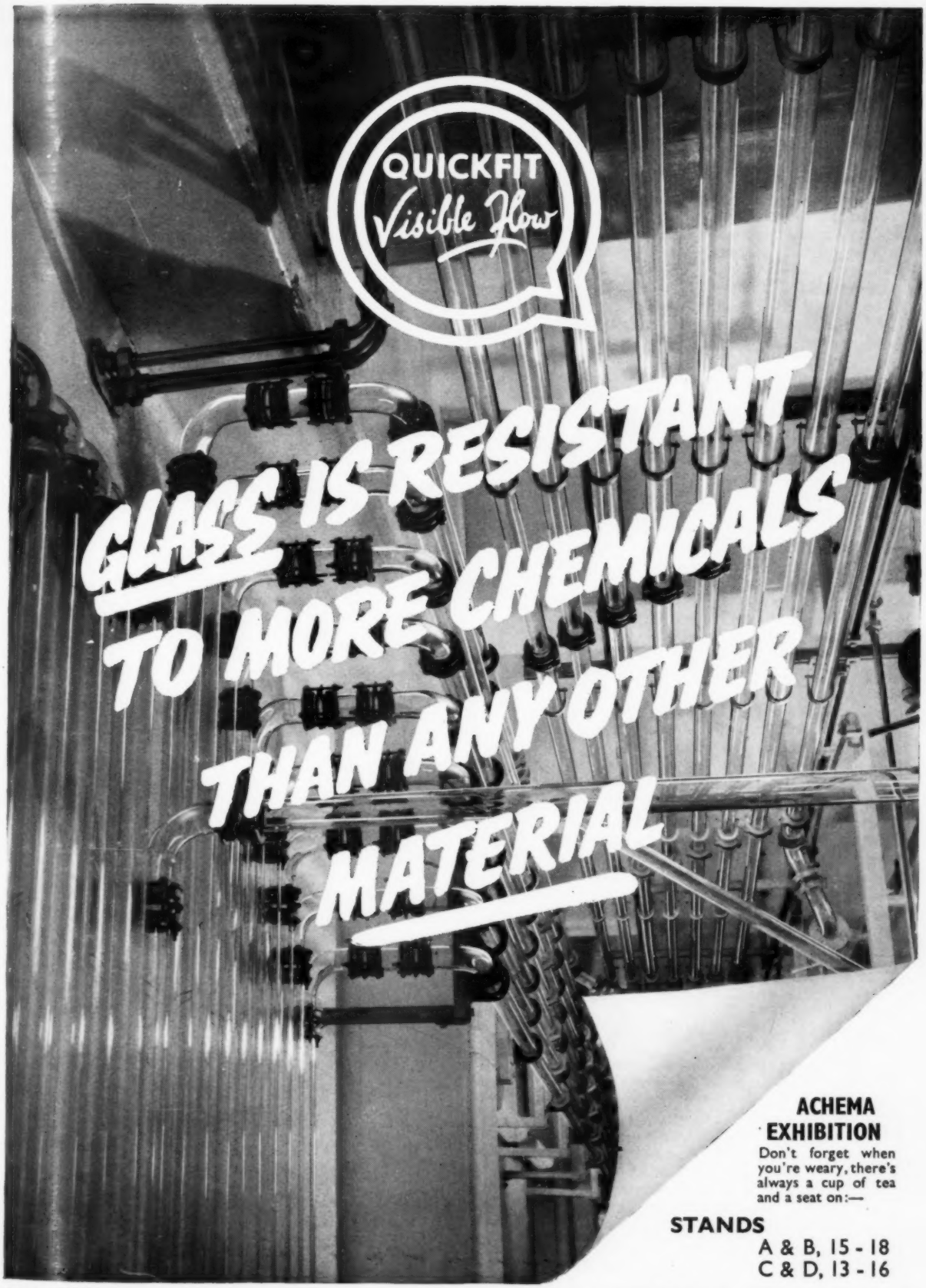
In practice, even a simplified system separating water and carbon dioxide from used space-cabin air requires additional equipment. Laden with moisture and carbon dioxide, the expended air goes first to a separator, where recoverable water is removed. The waterless air and carbon dioxide then pass through a molecular sieve, which allows the carbon dioxide only to reach the reactor. The remaining air then proceeds to pick up its revitalising quota of oxygen from the electrolytic cell and is inhaled as good air.

Meanwhile, the carbon dioxide from the molecular sieve has joined the hydrogen from the electrolytic cell in the Sabatier reactor and the resulting methane-moisture mixture goes into another separator, where the water is recovered to replenish the electrolytic cell.

Still more complicated is the overall sealed-cabin life support system. With longer flights, respiratory support systems become more complex and more dependent on other factors, such as heat dissipating radiators, power sources, structures and propulsion methods.

In the Boeing version of Sabatier process reactor, one ounce of small cylindrical pure nickel pellets are required to support the oxygen recovery system for one man. A test reactor, continuously producing water and methane for 12 days, has indicated that the catalyst possesses a satisfactory active life. It is thought that pair of reactors would eliminate the need for carrying an extra supply of the catalyst, since, while one system is being cleaned, the other could be in use.

Man, himself, is however a variable quantity to be reckoned with biologically with somewhat unpredictable inputs and outputs. Fed on algae, for example, he is estimated to utilise only half of it. When fed with air, his production of carbon dioxide varies according to diet, exercise, gain or loss in weight and his over-all condition. Were man an ideally balanced respiratory system, his intake of 2 lb of oxygen a day would yield exactly $1\frac{1}{4}$ lb of carbon dioxide, but this doesn't happen and a variable quantity has to be allowed for in calculations. Man, however, does produce from his own system $\frac{9}{10}$ lb of water a day which can be used by the electrolytic cell.



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Beet Sugar

THE transition from laboratory experiments to large-scale industrial development within a short period is in no case better illustrated than in the rapid rise of beet-sugar from a point 150 years ago. The chemist MARGGRAF had extracted sugar from beet by use of alcohol and had published papers on a potential industry based on beet. Yet it was left to his pupil ACHARD to publish in 1811 a Complete Treatise on the Beet-Sugar Industry, this changing the entire outlook when translated by the French chemist DEROSNE. ACHARD had a sponsor FREDERICK WILLIAM III of Prussia and had struggled to found a German industry at Kunern. Yet it was in France that a vigorous industry was created in the year of ACHARD'S publication; for NAPOLEON III caused 70,000 acres of beet to be planted, the French government appropriated a million francs for developing the industry, and very soon forty small factories were in operation. Though the Napoleonic wars, which had catalysed this activity when cane-sugar imports were cut off, soon came to an end, the impetus of ACHARD'S paper and a spate of technological discoveries proved sufficient to maintain progress.

A first example came from a pharmacist FIGUIER who discovered animal charcoal far superior to wood charcoal for decolorising sugar solutions, FIGUIER had been preparing boot blacking from a curious mixture of honey and bone charcoal and noted the decolorising effect, while a second Frenchman GUILLON saw animal charcoal thrown to waste in a Paris factory and also tried it as "bleaching" agent for sugar. A second advance came with GOTTLING'S diffusion process for separating sugar from non-crystallisable matter by enabling the cell-walls of beet to act as membranes. Modern diffusion batteries replacing the old rasping down of beet, bagging the pulp and compressing the mass, sprang from this idea of GOTTLING. A year later came a third vital improvement when the HON. EDWARD CHARLES HOWARD of the ducal house of Norfolk introduced his vacuum pan. HOWARD'S invention was one of few master patents complete and successfful from its first appearance. HOWARD incidentally was well connected with the sugar industry, for he married the daughter of a London refiner and was the originator of the use of a suspension of aluminium hydroxide as a defecant for sugar solutions. In his vacuum pan he used a vacuum working with not more than an inch of mercury pressure, the pan having a double bottom heating surface. Although the chemical bleaching of sugar was discovered also in that productive period of 1½ centuries ago, the use of sulphur dioxide had to wait some years before general adoption despite the work of DRAPIEZ. In five years from 1811 no less than 213 small beet-sugar factories set up in France were evidence of the effect of ACHARD'S classic review.

Expanding Seaweed Industry

PROSPECTS for the Scottish seaweed industry seem bright in spite of increasing competition from overseas.* During the past 15 months there have been spectacular developments in the retailing of seaweed manure; sales of seaweed meal to compounders of animal foods and to farmers are steadily growing; and the production of alginates—the most important outlet for seaweed—is being expanded to keep pace with increasing home and exports sales. Textiles printers and papermakers have become major consumers of alginates, and a range of quick-setting jellies and milk-sets based on alginates has been introduced.

The chemicals industry are finding more commercial uses for mannitol, at present available as a by-product of the manufacture of sorbitol, as a chemical intermediate. It has possible uses in nylon-like polymers. The indications are that demand for mannitol may in future exceed the supply from the sorbitol process, and the manufacture of mannitol from seaweed may then become attractive.

Overseas, the Irish seaweed meal industry has expanded, and some sodium alginate has been produced. Output has also increased in Norway, where there are now about 40 producers, and seaweed meal has also been produced in Iceland. New organisations in Holland and Australia are to produce alginates, and a new South African firm has announced plans for making pharmaceutical products from seaweed. The technology of industrial products from seaweed is being studied at the University of Sydney and at the New Bedford Institute of Textiles and Technology, Massachusetts. Chemical work on seaweed and its industrial utilisation forms part of the programme of the marine laboratory at Chubut, Argentina.

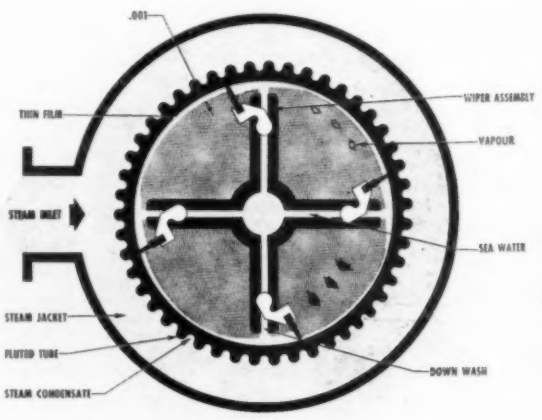
* Institute for Seaweed Research. Annual Report for 1960.

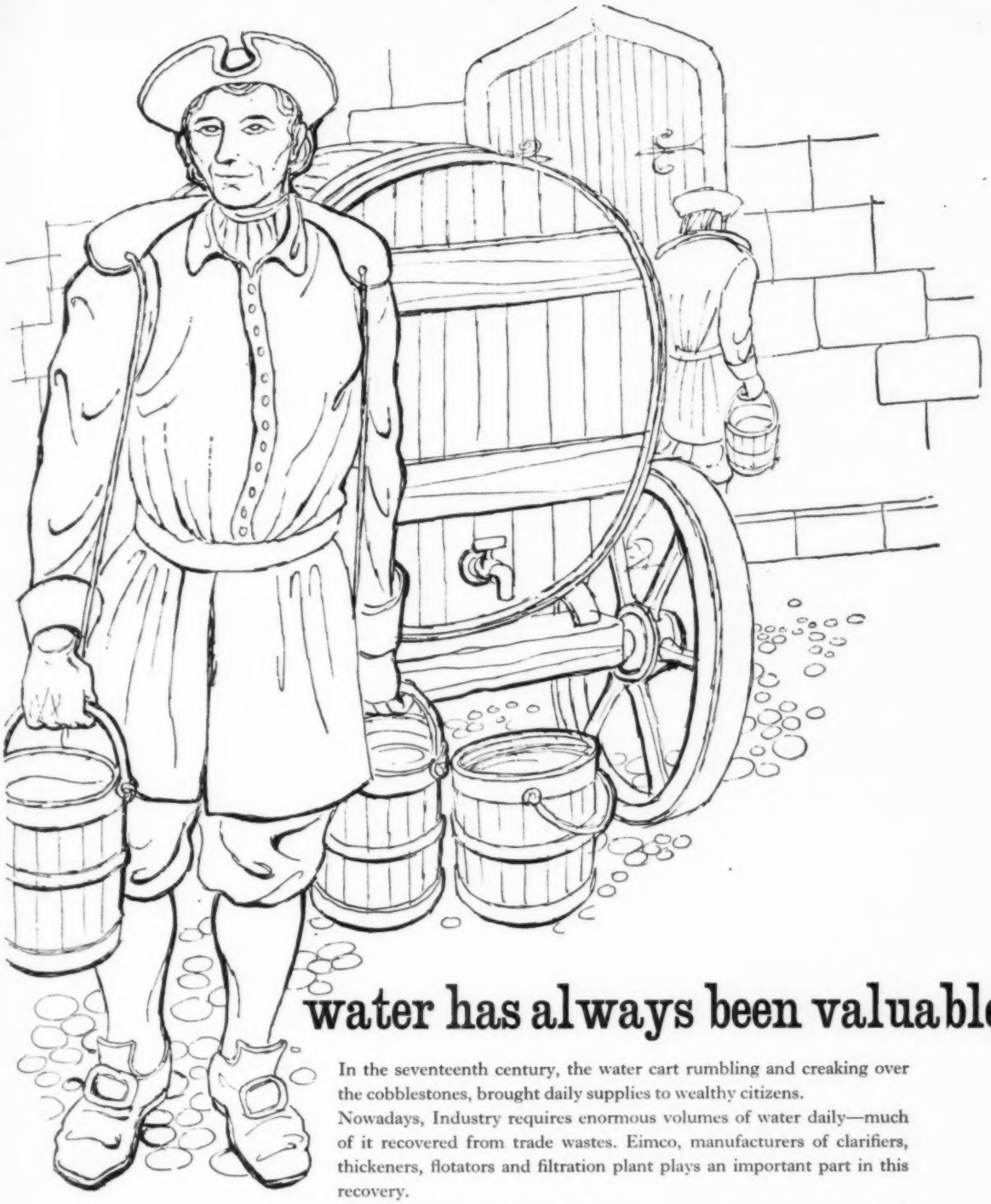
Thin-Film Salt Water Distillation Process

HIGH product purity and exceptionally high heat transfer rates are claimed for a new non-bubbling, non-boiling "thin-film" distillation process for preparing fresh water from brackish or sea water. The process, which has successfully completed a 1000 hr test in the laboratory, has been developed by the American General Electric Company, at Schenectady, under the sponsorship of the Office of Saline Water of the Department of the Interior and the Bureau of Ships of the Department of the Navy.

The main components of the G.E. experimental "thin-film" distillation system are a heat-transfer surface, a water distributor, and a rotating wiper assembly. The heat-transfer surface, upon which water is evaporated and condensed, is a 6 in diameter pure copper tube, 50 in long and with a wall thickness of 0.05 in. The outer surface is fluted. The 6 in copper tube is surrounded by a concentric 10 in diameter heat-resistant glass observation cylinder which in turn is surrounded by a 14 in diameter steel support tube. (A production distilling unit would employ a 10 in diameter metal outer steam jacket tube in place of the glass tube and supporting structure used in the laboratory system.)

Inside the heat transfer tube is a wiper assembly which rotates at 200 RPM about the vertical axis of the cylinder. Sea water feed enters the distilling unit through the centre of the base and flows upwards through the hollow wiper shaft. At the top of the shaft, a water distributor discharges the sea water at four points on the inside wall of the inside tube, just in advance of each set of wiper blades, which spread it over the entire inner surface of the cylinder. As





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the thin film evaporates, the vapour formed is drawn off at the top of the tube. Surplus feed water flows by gravity ahead of the wiper blades and is removed as a concentrated brine solution at the base of the unit. The absence of bubbling in the thin film minimizes brine carry-over and as a result, a high purity condensate is produced.

The system is able to extract approxmately 42 lb of fresh distillate from every 100 lb of sea water, with a salt-free purity of one part of salt per million parts of water. An overall heat transfer coefficient of 2500 Btu/hr/sq ft/°F has been achieved, and the apparatus, even in its experimental form, is light in weight.

General Electric are now plannng to deliver a 1300 gallon per day single-tube thin film water distillation unit to the United States Navy for a 1000 hr service evaluation test.

Uperisation

ALTHOUGH not one of the fields of prime interest to chemical engineers, the field of liquid sterilization is far from being devoid either of useful and relevent experience, or of scope for use of tools of the trade.

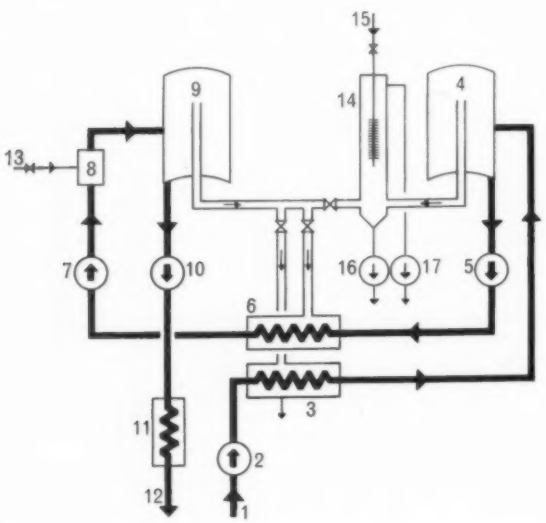
A process of recent inception* serves to illustrate this point. The aim of this process is the sterilization of liquids requiring treatment without detriment to their essential qualities; an extremely rapid rate of heating is demanded; this required a thorough study of the condensation of steam in a flowing liquid in order to evolve a design of injector which would provide uniform distribution of heat as well as the most rapid elevation of temperature. Once the necessary temperature has been attained it is necessary for an equally rapid cooling to be applied to the liquid under treatment.

Milk and dairy produce were the first liquids to be treated in this way and one of the outstanding advantages has been the fact that milk that has undergone "Uperisation", as the process is called, can be kept in a sterile container at room temperature for long periods.

The diagram illustrates the working principle of the process. The milk is first passed through a pre-heater 3 (with vapour heating) and is then de-aerated in vessel 4, in which pump 17 maintains a vacuum. The vapour removed at this point is liquefied in condenser 14 and led off by a condensate pump 16. The de-aerated milk is now further preheated in heat exchanger 6 and then goes to the Uperisation unit proper, 8, usually passing on the way through a homogenizer. In unit 8 the milk is rapidly heated to Uperisation temperature by steam injection, is then kept for a short time at this temperature (while it flows through a suitable length of pipe) and is finally cooled in expansion vessel 9. In most cases it then flows to a further cooler 11 and is subsequently filled into containers. The vapour produced by the expansion of the milk is used for the preheating stages.

The process is not restricted to dairy products. Fruit juices, sugar solutions, gelatine, honey and liquids with solids in suspension have been treated in Uperisation plants.

* Developed jointly by Sulzer Bros. and Alpura A.G. of Berne.



Improving Fuel Element Heat Transfer by Surface Roughening

WHEN fuel element canning materials such as, beryllium, stainless steel and other high temperature materials are used, fins are not a practical proposition for improving heat transfer. An alternative is to subdivide the fuel to give a large primary surface, but for various reasons it is not desirable that primary surface area should be unduly large. With a view to overcoming these difficulties, a basic investigation of heat transfer and friction for surfaces having various sizes and shapes of roughness has been carried out at Windscale, followed by an examination of their potential applications to highly rated gas-cooled reactor fuel elements.

Since roughness in the form of circumferential ribs has the advantage of conferring greatly enhanced resistance to deformation under gas pressure differences, this type of surface has therefore been the one to be most closely examined. V. WALKER* who investigated the properties of a surface of this kind, reports that the friction factor and the Stanton number are functions of the relative rib height and pitch and of Reynolds number.

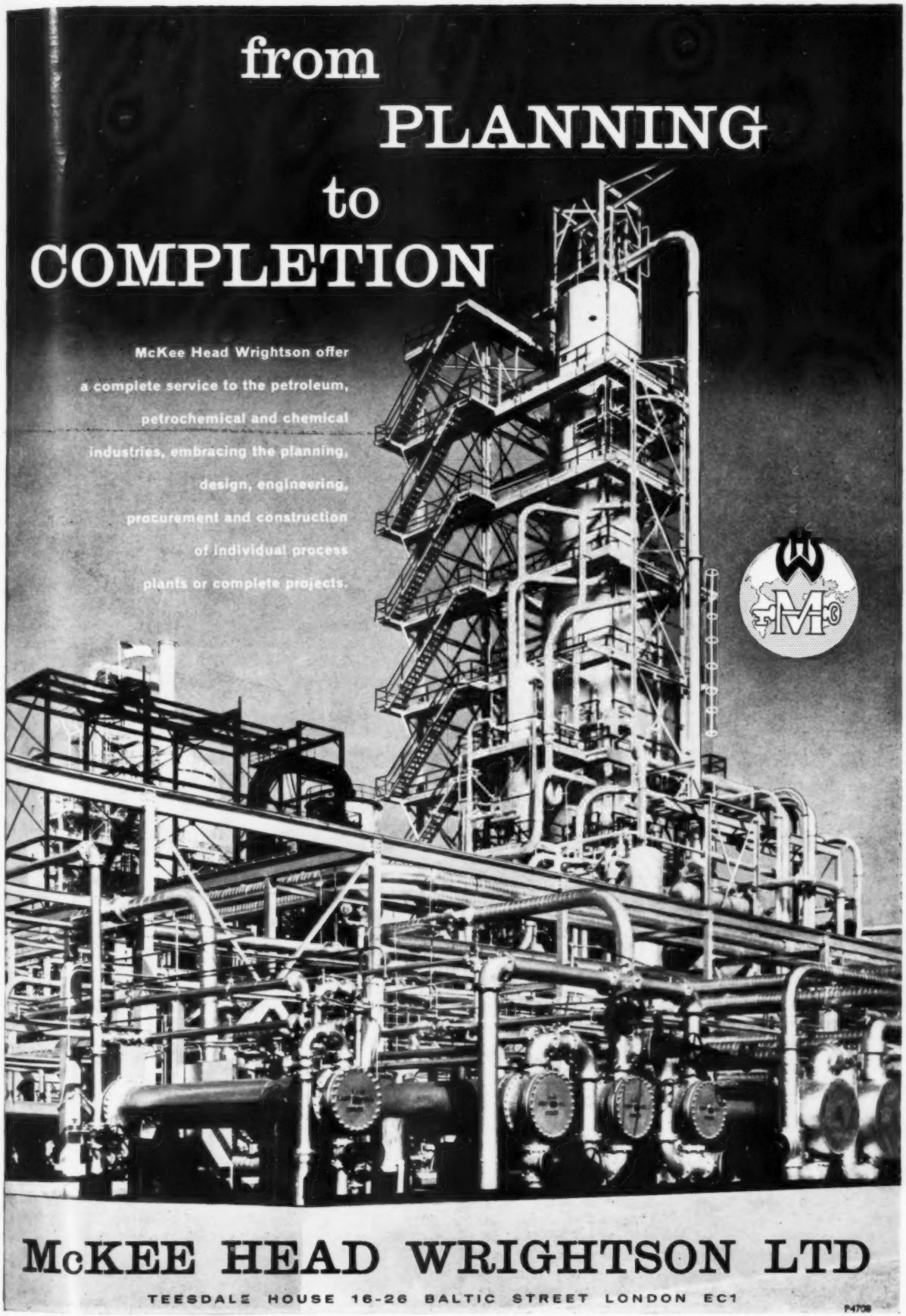
He has developed a number of graphical relationships representing the performance of the surface and from his results he recalculates a fuel element assembly. It is shown that for a particular CO_2 cooled channel that would require 57 smooth rods per cluster and 1197 rods in all, if roughened rods were used, the same thermal performance could be achieved using only 17 rods in a cluster and 204 rods in all—and these calculations involve pessimistic assumptions on the frictional effects.

* V. Walker, "Nuclear Engineering", April 1961, p. 144

Controlled Fusion Research

THE achievement of confining a stable deuterium plasma at a temperature of 30 million deg C for a period as long as 0.001 second is reported by the U.S. Atomic Energy Commission.* The plasma was heated by magnetic compression in two stages of the three-stage multiple compression magnetic mirror machine (Toy Top III) at the University of California's Lawrence Radiation Laboratory, Livermore. This is regarded as a substantial step towards the accomplishment of controlled atomic fusion of light elements, in particular because it shows promise of overcoming the difficult problem of plasma instability within the magnetic field. In the Livermore experiment, the time of confinement appears to be limited only by the period during which the pulsed confining magnetic field could be maintained at a sufficiently high value.

The mean energy of the ions was about 3000 electrons volts. (The ideal ignition temperature for a deuterium-tritium reaction is 5000 electron volts, and for a deuterium-deuterium reaction about 40,000 electron volts). The density of the ions was about 20 x 10^{12} per cu centi-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metre and the plasma volume about 2 litres. Neutrons were produced by deuterium-deuterium reactions during the entire period of confinement.

When the third stage of Toy Top III is brought into action, additional magnetic compression and heating will be available. Three-stage operation should provide a plasma in which the mean ion energy is increased to, possibly, three times the level obtained in the two-stage machine. This might be well into the range of temperature at which thermo-nuclear reactions can occur. If the machine succeeds in maintaining the stability of the higher temperature plasma, the next step would be to try to extend the period of plasma confinement. It is hoped to increase by severalfold the period during which the magnetic field strength is adequate to prevent loss of plasma.

AEC now see a distinct possibility of reaching the stage where they can assess the possibilities of achieving a fusion reactor that will yield more power than it takes to operate.

* Testimony by U.S. Atomic Energy Commision before Joint Committee on Atomic Energy, U.S. Congress, February 21, 1961.

Uranium Steels with Valuable Properties

LOW cost corrosion resistant steels with high endurance limits and capable of being used at high temperatures, such as are often required in chemical engineering, have been developed by the Physical Metallurgy Division of the Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada, by adding a small amount of uranium per ton of steel.

In short tests at 50°C. (122° F) in oxygenated solutions of nitric, sulphuric and hydrochloric acids, the uranium-containing steels exhibited less pitting and corrosion than the non-uranium-containing control steels. The table below illustrates the weight loss figures. This is a severe corrosion test, consequently the weight loss for all tests was high. Further testing is required to demonstrate whether the uranium-containing alloys are significantly better under service conditions. A chain has been placed on test, with other specimens under trial, in the Bay of Fundy to determine whether the pitting corrosion observations in the laboratory are duplicated under marine conditions.

Table 1. Corrosion of Uranium-Bearing 1040 Steels in Oxygenated Acids at 50 deg.C. (122 deg.F.).

Acid	Length of Test	Corrosion Rate, mils/year			
		0% U	0.07% U	0.12% U	0.23% U
14% HCL	10 min.	13,200	3600	8150	4900
14% HCL	do.	15,700	8000	9800	6950
10% H_2SO_4*	3 hrs.	16,000	3650	5900	3550
10% H_2SO_4	do.	14,800	3200	5850	3550
95-98%	do. 40 hrs.	112	118	108	97
70% HNO_3	10 hrs.	910	500	535	660

* In contact with argon instead of oxygen.

Uranium increases the fatigue strength of plain carbon steels. Uranium-free steel containing 0.40 per cent carbon has an endurance limit of 29,000 psi, whereas 0.02 per cent uranium and 0.20 per cent uranium additions to the 0.40 per cent carbon steel increase the endurance limit to 36,000 and 40,000 psi, respectively. A small quantity of uranium can produce a large initial increase in the fatigue strength of the material.

Stress rupture tests of SAE 1010 and SAE 1040 steels show an increase in time to rupture at a given stress and temperature with uranium added. Similarly, it has been found that increasing carbon at a constant uranium level improves the rupture life. Without uranium, failure in SAE 1010 steel occurs almost immediately, whereas uranium additions produce very significant increases. This feature should be of interest in steels for high temperature nuts and bolts, high temperature steam piping and other items used at temperatures in the neighbourhood of 1000°F (540°C.)

Work is also being done in connection with the addition of uranium to cast iron and copper-, aluminium-, magnesium- and zinc-based alloys.

Recovering Solutes by Foam Separation

MOST engineers, given the task of recovering trace quantities of soluble matter from solutions, would probably think first in terms of conventional ion exchange, adsorption, evaporative or membrane techniques. These however are by no means the only ones in the field as the recently announced "Foamet"* process shows.

This process is based on surface activity phenomena. A surface-active foaming agent that will combine specifically with the ion to be recovered is added to the solution. The surface-active material then concentrates in a molecular layer at the surface. Continuous generation of new foam causes the material concentrated on the foam surface to flow forward to a separate recovery unit.

In the pilot plant now in operation, the foaming agent is added to the feed before pumping it into the top of an 8 ft high section of the 10 ft counter flow column. Air or other gas enters through a sparger at the bottom of the pool and bubbles to the surface, forming foam, which is pushed up the column counter-current to the descending feed liquid. As the liquid drains over the foam bubbles, surface-active material concentrates at the bubble surfaces.

At the top of the countercurrent section, the column enlarges from 6 in to 15 in diameter. The foam discharges through a take-off at the top and is ducted to a centrifuge which breaks the foam. As little as one part in a hundred of the feed liquid discharges as enriched foamate liquid. The depleted bulk liquid is discharged from the pool at the bottom of the column.

As to performance, so far it has been found possible to reduce the concentration of radioactive Sr 90 and Cs 137 in low-level nuclear waste from 10^{-5} to 10^{-12} mol/mol using three foam columns in countercurrent series. It has also been used successfully for purifying solutions containing various metallic impurities; for recovering radium, vanadium, rare earths and uranium; for removing calcium and magnesium from sea water; and for purifying water itself.

* Developed by Radiation Applications, Inc., Long Island City, N.Y.

DIARY

Institution of Chemical Engineers

May 30. Royal Commonwealth Society, Craven Street, London, W.C.2 (Craven Street runs from the Strand to the Victoria Embankment adjacent to Charing Cross Station). **2.30 p.m.** Symposium on Biochemical Engineering (see opposite page for further details).

Institute of Petroleum

June 7-10. Old Swan Hotel, Harrogate. "Information and Dissemination" 6 Papers to be presented.

Exhibitions

June 9-17. Achema Congress and Exhibition, Frankfurt.

BIOCHEMICAL ENGINEERING SYMPOSIUM

THE practice of fermentation until fairly recently has been confined mainly to the manufacture of fermented beverages, the leavening of dough and the production of such foods as cheese and sauerkraut; and ethyl alcohol was one of the few fermentation products of importance. Later the manufacture of acetone and butanol by fermentative means was developed, to be eventually supplanted by synthetic processes.

More recently a stimulus to intensive fermentation has been provided by the discovery of such antibiotics as penicillin and streptomycin and a number of other antibiotics and pharmaceutical chemicals. These newer processes gave rise to problems quite different from those encountered in the older fermentation processes, and as a consequence chemical engineers became much more deeply involved in this field than was previously the case. This indeed gave rise to the term "Biochemical Engineering" upon which subject the first paper given to the Institution of Chemical Engineers was published more than seven years ago. This was presented by J. J. HASTINGS and dealt with a number of problems in the manufacture of antibiotics and vitamins. Since that time the occasional paper has appeared dealing with detailed aspects of the field; of these, two by P. H. CALDERBANK came to mind, on the subject of "Physical Rate Processes in Industrial Fermentation."

The time has now arrived when it is possible for the first symposium on the subject to be held by the Institution of Chemical Engineers; this will take place this month in London on Tuesday, 30th May, 1961, at the Royal Commonwealth Society, Craven Street, W.C.2.

The meeting will begin at 2.30 p.m. and will continue until approximately 6.45 p.m. There will be ample opportunity for discussion of the six papers to be presented.

Afternoon tea will be served free of charge to all those registering to attend.

Registration fees will be payable as follows:—

Student members of The Institution of Chemical Engineers ... ... 5s.

Graduate and Corporate members of The Institution of Chemical Engineers ... ... 10s.

Non-members of The Institution of Chemical Engineers ... ... 20s.

The registration fees include the provision of advance copies of the papers to be presented. Those not wishing to attend the symposium may obtain advance copies on payment of the appropriate registration fee.

The papers to be presented are:—

2.35 p.m. Developments in Fermentation by D. J. D. HOCKENHULL (Glaxo Laboratories Ltd.).

2.50 p.m. A rate equation for the heat sterilization of air by R. ELSWORTH, E. J. MORRIS and D. N. EAST (Microbiological Research Establishment, Porton).

3.05 p.m. Discussion.

4.30 p.m. Physical rate processes in industrial fermentation Part III: Mass transfer from fluids to solid particles suspended in mixing vessels by P. H. CALDERBANK and S. J. R. JONES (Warren Spring Laboratory, D.S.I.R.).

4.45 p.m. Aeration processes for the biological oxidation of liquid wastes by A. L. DOWNING and R. W. BAYLEY (Water Pollution Research Laboratory, D.S.I.R.).

5.00 p.m. Equipment for Laboratory and Pilot Plant Scale Fermentations by A. PARKER, E. P. MCCANN, D. PICKLES and D. G. WRIGHT (I.C.I. Pharmaceuticals Division).

5.15 p.m. Aeration and agitation in 5-litre fermenters by N. BLAKEBROUGH and M. W. WALTER (Chemical Engineering Department, University of Birmingham).

5.30 p.m. Discussion.

6.45 p.m. End of Symposium.

Further information and Registration Forms may be obtained from The Institution of Chemical Engineers, 16 Belgrave Square, London, S.W.1. (Telephone BELgravia 3648). The closing date for registration is the 25th May.

PROFESSIONAL APPOINTMENTS

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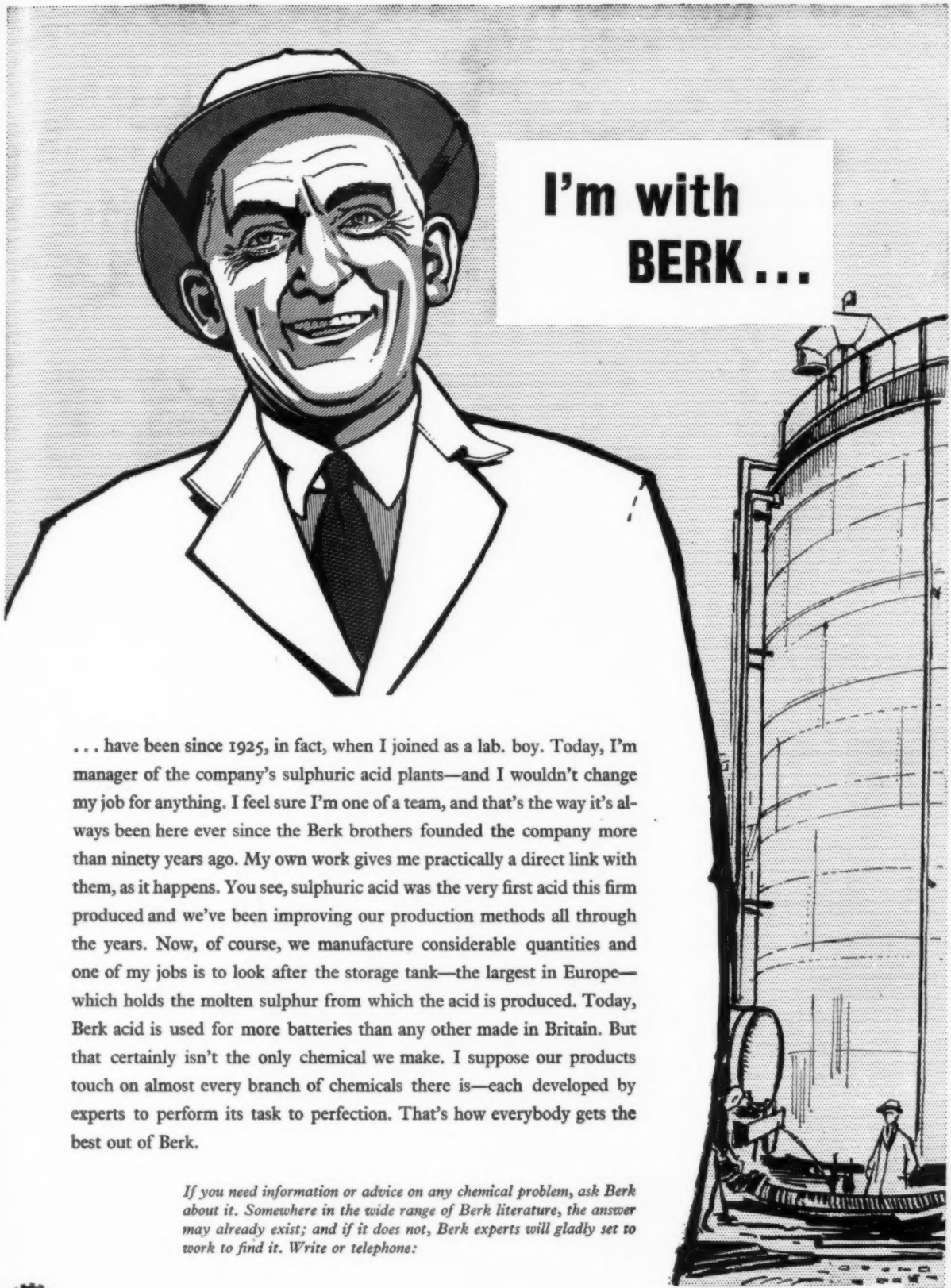

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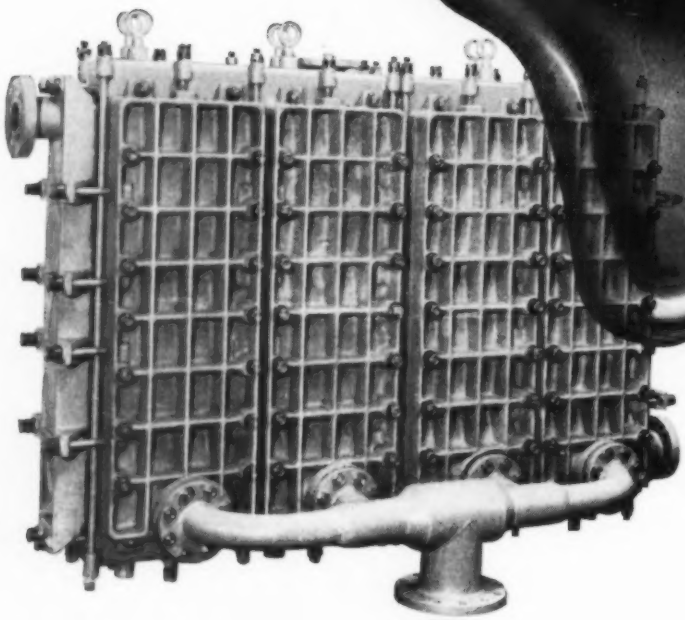

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Communication in Science and Industry

KNOWLEDGE, its acquisition, preparation and dissemination, pose problems for all who are active in any sphere of work possessing a scientific basis. Indeed, almost any engineer, technician or scientist would admit to the struggle required to keep abreast of the mass of invention, discovery and development with which he is continually confronted. Adding to his difficulties is the fact that some types of knowledge are becoming increasingly difficult to acquire. As one of the speakers at a recent D.S.I.R. conference on the subject of information, M. R. HOPKINS points out, the modern science student—but the remarks are of much wider application—has not only to be more familiar with a greater volume of knowledge than his predecessors, but he has to think in a more abstract way and to perceive subtler relations. In the nineteenth century to understand a physical situation meant to postulate a mechanism capable of producing its main features. Essentially the basis of understanding was a mechanical model, whereas today the readily understood mechanical model has been replaced by a mathematical one.

Apart from difficulties of gaining knowledge, there is the related question of its dissemination, a topic treated at the same conference by R. M. LODGE in relation to an industrial organization. He pointed out that much existing knowledge is missed and, whereas the number of discoveries is rapidly increasing, the rediscovery of what has been accomplished is increasing even more rapidly; this, indeed, indicating an alarming waste of effort.

Here it is worth recalling some of the astonishing revelations of the D.S.I.R. survey, published a few years ago, "The Use of Technical Literature by Industrial Technologists", and quoted by PERCY CUDLIPP at this conference. This survey, which covered over 1000 technologists from 127 establishments, reported that only 22 per cent stated that they would refer first to the literature if faced with a problem which could not be solved from memory or from customary sources of reference; equally amazing was the admission by some 88 per cent of the 700 technologists actually working on problems, that they commenced operations without taking the initial step of a literature search. Added to this, less than 40 per cent of them were aware of abstracts in their own particular field, only three-quarters who knew about abstracts made use of them, and a mere 20 per cent were able to remember abstracts which they had consulted in the last three months of their work.

The failure to be conscious of new discoveries can be ascribed to a disregard of the literature as well as to any material shortcoming in the means of making literature available, such as libraries. It may also be a reflection, of course, upon the value of much that appears in literature, and upon indifferent summaries which accompany some published works. A great deal of good work can be overlooked if summaries are couched in such vague terms as to obscure the main points of the work.

Further, the knotty problem of information retrieval is not helped by the voluminous literature and lack of uniformity in some aspects of presentation; some journals, for example, publish summaries, others do not: some classify their articles according to the Universal Decimal system, others use no system. However, the available subdivisions of the U.D.C. are scarcely appropriate for the many branches of chemical and other fields of engineering; what is needed is another system, perhaps using some of the ideas of the U.D.C., but which covers comprehensively the various branches of each domain independently of each, which would be adopted by all scientific and technical publications.

Returning to the narrower problem of the circulation and exchange of information within an organization, a number of telling points were made by LODGE, quite apart from the stress laid upon the value of teamwork. First, he suggests that the "rank-and-filers" should be told much more than the bare knowledge necessary to carry out their particular tasks within an organization. They should be provided with enough information to appreciate the importance of their work to the entire undertaking, for nothing is more depressing than to be working in ignorance of the reason for one's efforts. Again, it is useful, once an internal report has been prepared, to hold a meeting at which the report is passed over to other interested departments. LODGE likens this operation to the baton changing of runners in a relay race and aptly terms it "a hand-over meeting".

Two broader aspects of this general problem of knowledge may be mentioned, both of which are of national importance. First, the population as a whole needs to be much more aware of the place of science and technology in society; and this requires a reorientation on the part of the national Press, which is all too often concerned with the more dramatic and less pleasant aspects of science, to the exclusion of a balanced picture of its real function. The second aspect, a point made by W. S. ROBERTSON, another speaker at the D.S.I.R. conference, concerns Britain's at present inadequate information links with the Continent which prevent the arrival of a great deal of news of innovations produced on the Continent. In support of his case he draws attention to the efforts made by the Scots to restore their industrial position, a major line of attack being the creation of new international links through which technical information can flow, a factor which has contributed considerably to recent industrial progress in Scotland.

But when all is said and done, there is no escape from paper, and techniques of swift reading need to be developed.

D.S.I.R. Conference, "Problems of Communication in Science and Industry", held at Brangwyn Hall, Swansea, April 18 and 19, 1961.

622.731:621.926

Fig. 1. 84-in. Hydrocone crusher for tertiary crushing of Semi-Taconite ore (specular hematite). (Humboldt Mining Co., Humboldt, Michigan.)

CRUSHING & GRINDING CALCULATIONS PART I

The crushing and grinding of ores, rocks and minerals is an industrial process of great importance. Specialized engineering knowledge is required for the solution of practical problems in particle size reduction, and codification of this knowledge has hardly begun. The present paper is an attempt to assemble a highly condensed summary of the principal calculation methods which the author has found useful. References are given to articles with a more extensive explanation and examples of calculations.

by **FRED C. BOND**

COMMINUTION theory is concerned with the relationship between energy input and the product particle size made from a given feed size. It continues to be a rich field of controversy.

The oldest theory (1867) is that of RITTINGER,[1] and it still has adherents. He stated that the area of the new surface produced by crushing or grinding is directly proportional to the useful work input. The surface area of a ton of particles of uniform diameter d is proportional to $1/d$, and according to RITTINGER the useful work input per ton is also proportional to $1/d$. However, the measured surface energy of the new surface area produced is only a very small fraction of the order of 1/1000 of the energy input actually required to produce that surface in commercial crushing and grinding. Nearly all of the required energy input appears as heat after the particles are broken.

The second theory (1885) is that of KICK.[2] He stated that the work required is proportional to the reduction in volume of the particles concerned. Where f is the diameter of the feed particles and p is the diameter of the product particles, the reduction ratio Rr is f/p. According to KICK, the work input required per ton is proportional to $\log Rr/\log 2$.

Since neither theory agrees with commercial crushing and grinding results, the author developed the Third Theory in 1951.[3] According to this theory, the work input is proportional to the new crack tip length produced in particle breakage, and equals the work represented by the product minus that represented by the feed. In particles of similar shape, the crack tip length is equivalent to the square root of one-half the surface area, and the new crack length is proportional to $1/\sqrt{p} - 1/\sqrt{f}$.

For practical calculations the size in microns which 80 per cent passes is selected as the criterion of particle size. The diameter in microns which 80 per cent of the product passes is designated as P, the size which 80 per cent of the feed passes is designated as F, and the work input in kilowatt hours per short ton is W. The basic Third Theory equation is:

$$W = \frac{10\ Wi}{\sqrt{P}} - \frac{10\ Wi}{\sqrt{F}} \qquad \ldots.(1)$$

where Wi is the work index. The work index is the comminution parameter which expresses the resistance of the material to crushing and grinding. Numerically the work index is the kWh per short ton required to reduce the material from theoretically infinite feed size to 80 per cent passing 100 microns, equivalent to about 67 per cent passing 200 mesh. When any three of the quantities in Equation (1) are known, the fourth can be found by transposing the equation. Useful forms are shown in (1a) and (1b) below:

$$Wi = W \Big/ \left(\frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}} \right) \qquad \ldots.(1a)$$

$$P = \left(\frac{10\ Wi \sqrt{F}}{W\sqrt{F} + 10\ Wi} \right)^2 \qquad \ldots.(1b)$$

The work input in joules or watt-seconds per gram equals 3.97 W.

If the material is homogeneous to size reduction, its Wi value will continue constant for all size reduction stages.

FRED C. BOND is the Senior Staff Engineer in the Process Machinery Department, Allis-Chalmers Manufacturing Co., Milwaukee, U.S.A. He is a member of A.I.M.E. and served as chairman of the Committee on Comminution Research from 1948 to 1951.

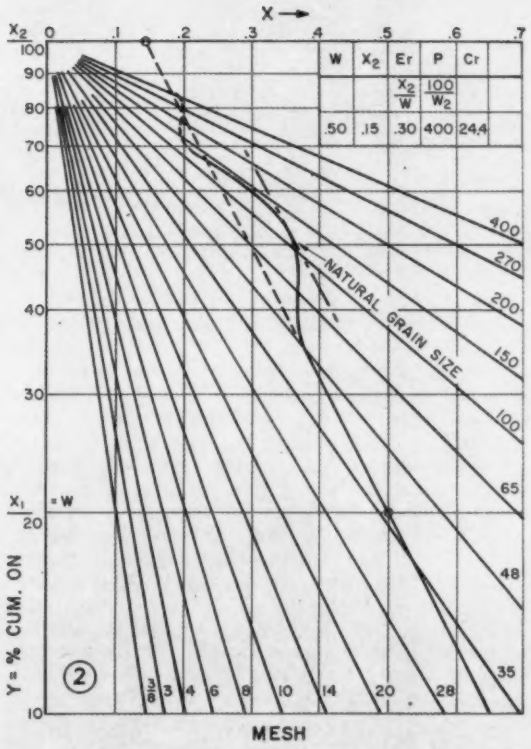
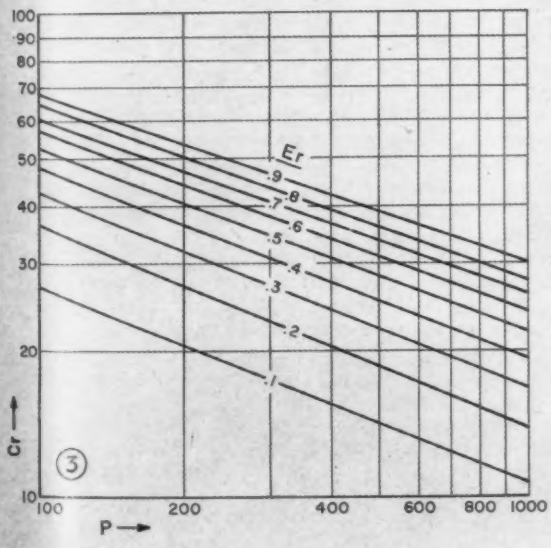


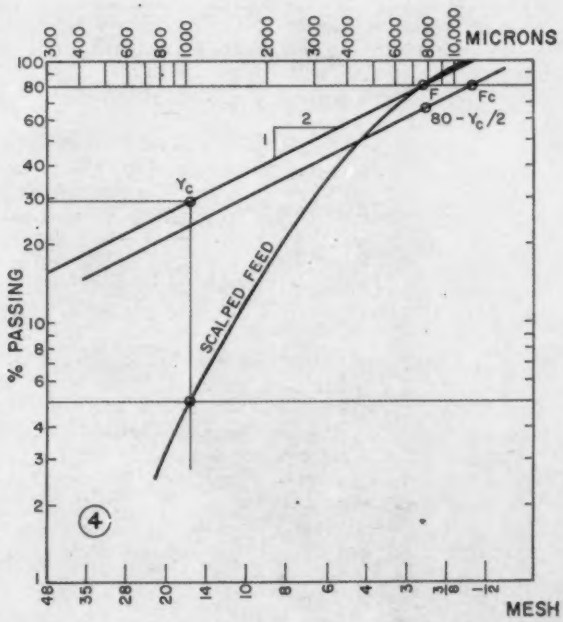
Fig. 2. Exponential size distribution plot of ore with natural grain size between 100 and 150 mesh. Exposure ratio Er = 0.30, P = 80 *per cent passing 400 microns. Crack length equals* Cr = 24.4 *cm/cc of solid.*

Fig. 3. Crack length plot from Table I—from 80 per cent passing 100 microns to 80 per cent passing 1000 microns for all values of exposure ratio Er.

Fig. 4. Scalped feed correction plot. Scalped feed with 80 per cent passing 7900 microns (F = 7900). Y = 29 *per cent,* 80 − Y/2 = 65.5 *per cent. Slope* = 1:2. *Corrected feed size* Fc = 12,000 *microns.*



CRACK LENGTH PLOT



SCALPED FEED CORRECTION PLOT

However, heterogeneous structures in rock are common. For instance, certain materials have a natural grain size, and their *Wi* values will be larger below that size than above it. A loosely cemented sandstone of 48-mesh silica grains will have a larger *Wi* for a product with broken particles finer than 48 mesh than for a coarser product.

The efficiency of the reduction machine may also influence the operating work index. For instance, a ball mill grinding an ore from 80 per cent—14 mesh to 80 per cent—100 mesh will have a lower operating *Wi* value with 1.5-in. grinding balls than with oversize 3-in. balls.

A material may have an *induced* grain size resulting from some preferential sizing action which changes its natural size distribution. Undersize grinding balls can have this effect.

Laboratory determinations of the work index show the resistance to breakage at the size range tested, and any variation in the *Wi* values in tests at different product sizes shows that the material is not homogeneous to size reduction. For this reason laboratory tests should preferably be made at or near the product size required in commercial grinding.

The operating work index from transposed Equation (1a) can be calculated from size reduction in commercial plants to compare the plant efficiency with laboratory test results, to compare efficiencies of the different plant size reduction stages, or to compare the efficiencies of different plants treating similar materials. The work index is particularly valuable in predicting the size and capacity of new installations. Table IIIA in the appendix lists the average work index values of 82 different materials.

Three Principles

Comminution phenomena have recently been redacted into three principles,[4] which are useful guides for the consideration of all crushing and grinding data.

The *First Principle* states that, since energy input is necessary for size reduction, all feed particles of finite size have a certain energy register, or energy level, which must be added to the energy input during crushing or grinding to obtain the energy register of the product. All statements of the energy utilized in comminution must satisfy this condition:

energy input = energy register of product − energy register of feed.

The Third Theory work index Equation (1) follows this principle, with the energy register equal to the total specific energy input in kWh per short ton. The work index *Wi* is the energy register to 80 per cent passing 100 microns.

If the energy which has been expended in preparing the feed particles is neglected in analysing comminution data, the first principle has been violated, and application of the calculated result to different feed and product sizes will be distorted.

The *Second Principle* states that the useful work input in crushing and grinding is proportional to the length of the new crack tips produced. In ordinary crushing and grinding, rock particles absorb strain energy and are deformed under compression or shear until the weakest flaw in the particle fails with the formation of a crack tip. This minute change of shape causes other crack tips to form at other weak flaws, and the particle breaks, releasing the bulk of the strain energy as heat. The strain energy required to break is proportional to the length of the crack tips formed, since the additional energy required to extend the crack tips to breakage is supplied by the flow of the surrounding resident strain energy to the crack tips.

Since the crack tip length is proportional to the square root of the new surface area produced, the specific work input required is inversely proportional to the square root of the product particle diameter minus that of the feed diameter, as shown in the work index Equation (1). Crushing and grinding machines are essentially devices for the conversion of mechanical energy into strain energy into heat, under conditions which promote material breakage.

The energy register as used herein represents the specific energy which has passed through the material as strain energy, and includes heat losses and losses due to friction and other causes. It does not correspond to the energy content of the material.

The *Third Principle* deals with the relationship of particle flaws to material breakage. A flaw is defined as any structural weakness in a particle which may develop into a crack tip under strain. Flaws are always present in brittle materials and may cause wide variations in the breaking strengths of apparently similar particles.

The weakest flaw in a particle determines its breaking strength in crushing and grinding. It also controls the number of particles produced by breakage. Particles with the weakest flaws break most easily and produce the largest product particles. However, they are not necessarily easier to grind to a given product size requiring several stages of breakage than are particles of the same size whose weakest flaw is stronger.

The Third Principle states that the weakest flaw in a particle determines its breaking strength but not its work index. The work index is controlled by the average flaw structure throughout the entire size range tested. Work index variations at different product sizes result from flaw concentrations or shortages at those sizes, usually caused by natural grain sizes.

Evaluation of Particle Size Distribution

The usual standard screen scale consists of a series of sieves with square openings differing by $\sqrt{2}$, based upon the 200-mesh sieve opening of 74.2 microns. There are 25,400 microns in an inch. A screen analysis size distribution of a crushed or ground product consists of a listing of the per cent weight passing or retained on each sieve in the series.

There is probably a definite law which governs the regular size distribution of crushed or ground products; however, none of the proposed laws has been generally accepted as yet.

Size distribution analyses of crushed and ground products are commonly plotted on log-log paper with (y) the per cent passing as ordinate and the particle diameter (x) in microns as abscissa. Such plots of complete samples usually show a fairly straight line for the finer particle size range which begins to curve in the coarser sizes and often approaches tangency with the 100 per cent passing line at the top of the plot. The size 80 per cent passes may be found from the curved portion of the plot for use in the work index Equation (1).

When the straight lower portion of the plotted line is extended at its slope α, it intercepts the 100 per cent passing line at k_{100} microns. It follows a *power* law defined by the GATES-GAUDIN-SCHUHMANN equation,[5] which is

$$y = 100 \left(\frac{x}{k_{100}}\right)^{\alpha} \qquad \ldots . (2)$$

From this equation the surface area Sc in cm² per gram of cubical particles of density ρ, with 100 per cent passing k_{100} microns and slope α to a grind limit of Li microns is:

$$Sc = \frac{60{,}000\ \alpha}{\rho\, k_{100}(1-\alpha)} \left[\left(\frac{k_{100}}{Li}\right)^{1-\alpha} - 1\right] \qquad \ldots . (3)$$

The grind limit Li has recently been assigned the value of 0.1 micron,[6] equivalent to 1000 Ångstrom units. This is about 200 times the unit space lattice of quartz and other rock forming minerals.

The slope α is often about 0.5, but may approach unity.

Crushing or grinding in closed circuit produces less fines than open-circuit operation, and causes α to increase. Removal of fines before reduction has the same effect. As a material is ground finer, its value of α often appears to decrease.

The log-log size distribution plot is convenient. However, the usual curvature in its upper part indicates that the actual size distribution law is of the exponential type with a variable exponent, rather than of the power type with the constant exponent α.

Exponential Size Distribution Plots

A method of plotting has been developed with yields size distribution lines that are apparently quite straight for homogeneous materials.[6] They follow the exponential equation.

$$Y = 100 - y = b/e^{aX} = b/10^{AX} \quad \ldots.(4)$$

$$AX = \log b - \log Y \quad \ldots.(4a)$$

X_1 represents w, the energy register in kWh/ton divided by the work index Wi at the 80 per cent passing base line where $Y = 20$. The per cent cumulative retained Y is $100 - y$, b is the $100 - y$ intercept, and A is the slope. On semi-log paper Y is measured on the vertical logarithmic scale, and X is on the horizontal linear scale. Diagonal straight lines are drawn radiating from the upper left-hand corner of the chart, which represent each mesh size on the $\sqrt{2}$ screen scale and cross the 80 per cent passing horizontal base line. Each diagonal line represents a mesh size testing sieve with an opening of P_1 microns, and crosses the base line at $w = 10/\sqrt{P_1}$. The diagonal lines can be assigned various mesh sizes, with the proper relationship between X_1 and w.

This plot is not as convenient as the log-log plot, but it has several advantages. The first is that the 80 per cent passing size P can be found with less error from $P = 100/w^2$, where w is the value of X_1 at the base line 20.

Another advantage is the delineation of natural or induced grain sizes. As the size distribution line proceeds up the chart approaching the finer sieve sizes, a curved loop to the right of the indicated straight line shows a grain size deficiency, culminating at the natural grain size where the loop becomes parallel to the straight line. The compensating grain size excess is shown by the return of the loop to the straight line. If laboratory determinations of the work index are made at the different sieve sizes, the low Wi values will increase as the grain deficiency sizes approach the natural grain size, and decrease at the grain excess sizes where the loop returns to the straight line.

The natural grain size in ores usually corresponds to the unlocking or mineral liberation size to which the ore must be ground before concentration. The exponential method of plotting the size distribution furnishes a very good indication of the unlocking size when the amount of the mineral to be concentrated is large. This is particularly true of iron ores, and the exponential plots show clearly the different unlocking properties of autogenous and conventional grinds.

Much additional information can be obtained from this type of size distribution plot, including crack length values.[6] In the ball mill grindability tests at 60 joules input per mill revolution, the joules required to produce 1 cm of new crack length in material of homogeneous breakage with specific gravity Sg is approximately $Wi\ Sg/11$.

The exposure ratio Er is related to b in the exponential size distribution Equation (6) as follows, where $Er = X_{100}/w$:

$$\log b = \frac{2 - 1.301\ Er}{1 - Er} \quad \ldots.(4b)$$

The data in Table I can be plotted on six sheets of single cycle log-log paper to make a set of charts from which the crack length Cr of any regular crushed or ground product can be found graphically when its 80 per cent passing size P and exposure ratio Er are known from an exponential size distribution plot. On the first sheet the Cr values for $P = 1$ micron are plotted on the left-hand side, and the values for $P = 10$ microns are plotted on the right-hand side. Each pair of points is connected by a straight line marked with its Er value, and intermediate lines can be drawn using a logarithmic rule. The second sheet is made by plotting values for $P = 10$ on the left-hand side and $P = 100$ on the right-hand side, and so on for the set of six charts, which cover the entire operating size range.

TABLE I—Crack Length Values for Plotting Cr (cm/cc)

P microns	1	10	100	1000	10,000	100,000	1,000,000
Er	Cr						
0.10	207.0	79.8	27.3	10.65	3.76	1.37	0.495
0.20	212.0	93.0	36.6	14.00	5.25	1.93	0.735
0.30	208.5	102.1	42.5	16.89	6.60	2.47	0.936
0.40	202.0	110.0	48.1	19.56	7.87	2.96	1.136
0.50	191.9	115.2	52.7	21.95	9.00	3.44	1.337
0.60	183.0	119.4	57.1	24.25	10.04	3.91	1.523
0.70	176.0	123.0	60.8	26.35	11.00	4.37	1.699
0.80	165.8	125.8	64.3	28.40	11.94	4.81	1.860
0.90	160.0	128.1	67.5	30.35	12.85	5.22	2.029

Surface Area Calculations of Ground Products

Approximate surface areas in sq. cm per gram of equivalent cubes for log-log size distribution plots can be calculated from Equation (3), using a grind limit Li of 0.10 micron.

When the crack length Cr has been found,[6] the surface of equivalent cubes in sq. cm per cc of solids is $2\ Cr^2$.

The WAGNER surface area Sw in sq. cm/gram is approximately equal to the BLAINE air permeability surface to the power 0.92, or $Sw = (B1)^{0.92}$.

The 80 per cent passing size P in microns has the following approximate relationship to the BLAINE and WAGNER surface areas:

$$P = \left(\frac{20{,}300}{B1}\right)^2 = \frac{3.63 \times 10^8}{(Sw)^{2.15}} \quad \ldots.(5)$$

and $\log P = 2 \log (20{,}300/B1) = 8.56 - 2.15 \log (Sw)$ $\ldots.(5a)$

Work Index from Laboratory Tests

Equations have been derived for finding the work index Wi from several types of laboratory crushability and grindability tests,[7] as described below.

Crushability Test

Pieces of broken rock passing a 3-in. square and retained on a 2-in. square are mounted between two opposing equal 30-lb weights which swing on wheels. When the wheels are released the weights strike simultaneously on opposite sides of the measured smallest dimension of the rock. The height of fall is successively increased until the rock breaks. The impact crushing strength in foot-pounds per inch of rock thickness is designated as C, and Sg is the specific gravity. The work index is found from the average of 10 breaks, where

$$Wi = 2.59\ C/Sg \quad \ldots.(6)$$

Rod Mill Grindability Test

The feed is crushed to $-\frac{1}{2}$ in., and 1250 cc packed in a graduated cylinder are weighed, screen analysed, and ground dry in closed circuit with 100 per cent circulating load in a 12 in. dia. by 24 in. long tilting rod mill with a wave-type lining and revolution counter, running at 46 rpm. The grinding charge consists of six 1.25 in. dia. and two 1.75 in. dia. steel rods 21 in. long and weighing 33,380 grams.

In order to equalize segregation at the mill ends, it is rotated level for eight revolutions, then tilted up 5° for one revolution, down 5° for another revolution, and returned

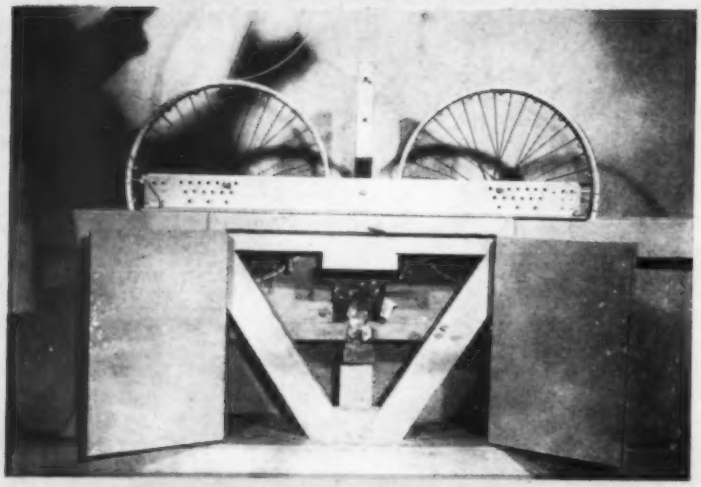


Fig. 5. Twin pendulums impact crushing device for measuring work index. Picture of specimen breakage taken at instant of impact.

to level for eight revolutions continuously throughout each grinding period.

Tests are made at all mesh sizes from 4 to 65 mesh. At the end of each grinding period the mill is discharged by tilting downward at 45° for 30 revolutions, and the product is screened on sieves of the mesh size tested. The sieve undersize is weighed, and fresh unsegregated feed is added to the oversize to make its total weight equal to that of the 1250 cc originally charged into the mill. This is returned to the mill and ground for the number of revolutions calculated to give a circulating load equal to the weight of the new feed added. The grinding period cycles are continued until the net grams of sieve undersize produced per revolution reaches equilibrium and reverses its direction of increase or decrease. Then the undersize product and circulating load are screen analysed, and the average of the last three net grams per revolution (*Grp*) is the rod mill grindability.

Where F is the size in microns which 80 per cent of the new rod mill feed passes, and P_1 is the opening of the sieve size tested in microns, then the rod mill work index Wi is calculated from the following revised (1960) equation:

$$Wi = 62/(P_1)^{0.23} \times (Grp)^{0.625} \times \left(\frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}}\right) \quad \ldots.(7)$$

This Wi value should conform with the motor output power to an average overflow rod mill of 8 ft interior diameter grinding wet in open circuit. For dry grinding the work input should be multiplied by 1.30. Where D is the mill diameter inside the lining in feet, the work input should be multiplied by $(8/D)^{0.20}$.

Ball Mill Grindability Test

The standard feed is prepared by stage crushing to all passing a 6 mesh sieve, but finer feed can be used when necessary. It is screen analysed and packed by shaking in a 1000-cc graduated cylinder, and the weight of 700 cc is placed in the mill and ground dry at 250 per cent circulating load. The mill is 12 in. × 12 in. with rounded corners, and a smooth lining except for a 4 in. × 8 in. hand hole door for charging. It has a revolution counter and runs at 70 rpm. The grinding charge consists of 285 iron balls weighing 20,125 grams. It consists of about 43 1.45-in. balls, 67 1.17-in. balls, 10 1-in. balls, 71 0.75-in. balls, and 94 0.61-in. balls with a calculated surface area of 842 sq. in.

Tests are made at all sieve sizes below 28 mesh. After the first grinding period of 100 revolutions, the mill is dumped, the ball charge is screened out, and the 700 cc of material is screened on sieves of the mesh size tested, with coarser protecting sieves if necessary. The undersize is weighed, and fresh unsegregated feed is added to the oversize to bring its weight back to that of the original charge. Then it is returned on to the balls in the mill and ground for the number of revolutions calculated to produce a 250 per cent circulating load, dumped and rescreened. The number of revolutions required is calculated from the results of the previous period to produce sieve undersize equal to 1/3.5 of the total charge in the mill.

The grinding period cycles are continued until the net grams of sieve undersize produced per mill revolution reaches equilibrium and reverses its direction of increase or decrease. Then the undersize product and circulating load are screen analysed, and the average of the last three net grams per revolution (*Gbp*) is the ball mill grindability.

When F is the size in microns which 80 per cent of the new ball mill feed passes, P is the microns which 80 per cent of the last cycle sieve undersize product passes, and P_1 is the opening in microns of the sieve size tested, then the ball mill work index Wi is calculated from the following revised (1960) equation:

$$Wi = 44.5/(P_1)^{0.23} \times (Gbp)^{0.82} \left(\frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}}\right) \quad \ldots.(8)$$

The average value of P at 100 mesh is 114 microns, at 150 mesh it is 76 microns, at 200 mesh it is 50, and at 325 mesh it is 26.7. These values of P are to be used in Equation (8) when P cannot be found from size distribution analyses.

The Wi value from Equation (8) should conform with the motor output power to an average overflow ball mill of 8 ft interior diameter grinding wet in closed circuit. For dry grinding the work input should normally be multiplied by 1.30. However, ball coating and packing can increase the work input in dry grinding.

Where D is the mill diameter inside the lining in ft, the work input should be multiplied by $(8/D)^{0.20}$.

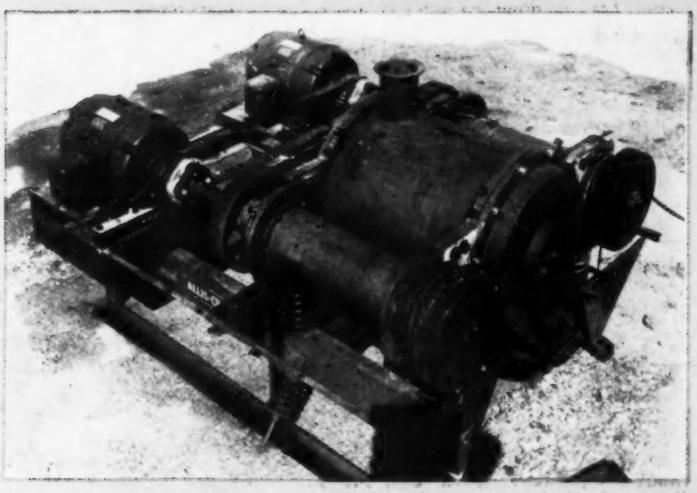


Fig. 6. 30-in. vibrating ball mill.

Hardgrove Grindability Rating

Where *Hd* represents the Hardgrove grindability rating,[13] the equivalent wet grinding work index is found from:

$$Wi = 435/(Hd)^{0.91} \quad \ldots . (9)$$

Crushing

Crushing is accomplished by contact with metal or other surfaces maintained in a fixed position or in a rigidly constrained motion path, although many crushers have safety features which allow release under excessive pressure. This is contrasted with grinding, which is accomplished by the free motion in response to gravity and other forces of unconnected media such as rods, balls, rock pieces and pebbles.

Free media grinding has several inherent advantages over fixed media crushing, and as reduction machinery increases in size and strength larger particles become amenable to grinding which could formerly be reduced only by crushing. Cases in point are the development of large peripheral discharge rod mills and autogenous grinding mills. However, the commercial production of particles larger than about $\frac{1}{4}$ in. is still a crushing process.

Crushing is usually done dry in several stages with small reduction ratios ranging from 3 to 6 in each stage. The machines used include: gyratory crushers, jaw crushers (both single and double toggle), crushing rolls, and impact crushers, hammer mills or pulverators. It is done with both screened and natural feeds, in stages with screens between each stage to remove undersize, as well as in open circuit and in closed circuit with screens.

Excessive moisture, fines, or both, in the feed can cause packing in the crusher, resulting in a decrease in capacity, increase in power drawn, and increase in the crushing plant work index. This is usually remedied by screening out more fines ahead of the crusher.

Crusher motor sizes are usually limited to protect the crushers against breakage. For the same reason uncrushable pieces of metal are usually removed from the feed magnetically, or the crusher is designed to open up and let them pass through.

Crusher Product Sizes

The crusher product size which 80 per cent passes at full capacity can be estimated from the crusher setting, eccentric throw and work index of the material.

The product sizes of jaw crushers and primary gyratory crushers with steep crushing cones are controlled principally by the open side setting of the crusher. Where *Oss* is the open side setting of the crusher in inches at the bottom of the crushing chamber, the 80 per cent passing size *P* of the crusher product in microns is calculated from Equation (10). 1 in. equals 25,400 microns.

$$P = (25{,}400)\ (Oss)\ (0.04Wi + 0.40) \quad \ldots . (10)$$

The product sizes of cone crushers, with their flat crushing cones and relatively high speeds, are controlled principally by the close side setting. Where *Css* is the close side setting of the cone crusher in inches at the bottom of the crushing chamber, as commonly determined by passing a piece of lead through the crusher, and *Ecc* is the eccentric throw in inches at the bottom of the crushing cone, the product size *P* is found from

$$P = \frac{(25{,}400)\ (Css)\ (7Ecc)\ (0.02Wi + 0.70)}{(7Ecc - 2Css)} \quad \ldots . (11)$$

If the material is very slabby, the value of *P* may be somewhat larger than that indicated by Equations (10) and (11). These equations are useful when screen analyses of the crusher products are not available.

Scalped Feed to Crushers

The Third Theory equations require a "natural" feed containing the natural fines produced in the previous reduction stages. When fines are removed from the feed, the relationship between *F* and *P* is altered. In most crushing installations where fines smaller than the crusher discharge opening are removed from the feed by screening, the work input per ton of original feed is not materially decreased, except as the removal of fines prevents the abnormal condition of packing in the crusher. It has been found satisfactory to disregard the scalping operation, and to consider the feed to the screen or grizzly as equivalent feed to the crusher. This is preferable in most cases where the grizzly separating size and hourly tonnage through are not known accurately.

However, in some instances where much of the fines have been removed the correction for scalped feed must be

made. This is done empirically by using that increased normal feed size *Fc* which is equivalent in work input per ton to the 80 per cent passing size *F* of the scalped feed. The per cent passing size distribution line of the scalped feed is plotted on log-log paper. A line with the normal slope of 1:2 is drawn through the 80 per cent passing point *F* to its intersection *Yc* with the size which 5 per cent of the scalped feed passes; a parallel line is drawn through the point with co-ordinates *F*, $(80 - Yc/2)$, *and its intersection* with the 80 per cent passing line gives the value of *Fc*.

When pieces all of one diameter of *d* microns are fed to a crusher the equivalent 80 per cent passing size *Fc* is that of a Third Theory size distribution line with an exposure ratio *Er* of 0.05 and the same crack length as the particles fed.[6] The crack length in cm/cc is $Cr = 173/\sqrt{d}$. The *Fc* values are listed in Table II. When the feed consists of particles of several different diameters *d* without fines, the equivalent corrected feed size *Fc* can be computed as the weighted average of the different sizes *d*.

TABLE II—Equivalent 80 per cent Passing Size *Fc* for Particles all *d* Microns Diameter. *Cr*=Crack Length in cm/cc=$173/\sqrt{d}$. *As*=Slope=$0.0699\sqrt{d}$.

Size		*As*	*Cr*	*Fc*
Sieve Inch Mesh	Microns d ($=P_1$)	Slope of sieve lines	cm/cc	Microns equivalent (*Er*=0.05)
3 in.	76,200	19.30	0.626	255,000
2 in.	50,800	15.73	0.769	163,000
1 in.	26,670	11.42	1.057	76,000
¾ in.	18,850	9.60	1.260	50,400
½ in.	13,330	8.56	1.500	34,000
⅜ in.	9,423	6.79	1.782	23,000
Mesh				
3	6,680	5.70	2.12	15,400
4	4,760	4.82	2.51	10,400
6	3,360	4.05	2.98	8,000
8	2,380	3.14	3.55	5,500
10	1,680	2.86	4.22	3,760
14	1,190	2.41	5.02	2,600
20	840	2.024	5.97	1,800
28	590	1.699	7.13	1,220
35	420	1.432	8.45	835
48	297	1.205	10.01	580
65	210	1.012	11.94	375
100	149	.853	14.18	250
150	105	.716	16.90	158
200	74.2	.602	20.02	103
270	52.5	.506	23.9	71
325	44	.464	26.1	59
400	37.1	.426	28.4	50
	30	.383	31.6	40
	25	.350	34.6	34
	20	.313	38.7	27.3
	15	.271	44.7	20.7
	10	.221	54.8	14.0
	5	.156	77.5	7.0

Grinding

Correction for Feed and Products

Closed-circuit grinding and complex grinding circuits which include concentration and separation equipment are best analysed by considering each circuit as an integrated unit. In closed-circuit grinding the unit consists of the mill and the classifier, with a single feed to the mill or classifier and a single classifier undersize product. Calculations from the mill discharge and circulating load are usually unsatisfactory because the harder fraction of the material accumulates, and the circulating load has an unknown higher work index than the new feed. If the closed circuit includes concentrating equipment such as magnetic separators which reject a tailing, the product of the grinding circuit is a calculated composite of the classifier undersize and the separator tailing, which should always be screen analysed for this purpose.

Calculations involving unnatural feed from which part or all of the fines have been removed should be avoided whenever calculations of integrated circuits can be substituted. However, the empirical methods described under "Scalped Feed to Crushers" can be used when necessary in grinding circuits.

When grinding tests are made in a small, continuous, pilot-plant mill of diameter *D*, the gross power input per ton ground should be multiplied by $(D/8)^{0.20}$ to find the work index from Equation (1a).

Fineness Adjustment

Experience has shown that extremely fine grinding requires additional energy input beyond that indicated by the work index Equation (1). There are several possible reasons for this, including:

(a) The ball sizes customarily employed are too large for extremely fine particles.
(b) In fine grinding the closed-circuit classification is usually either inefficient or absent.
(c) In dry grinding of fine particles the amount of ball coating ranges from incipient to complete. Ball coating cushions the metal contacts and decreases grinding efficiency.
(d) In wet grinding a thick viscous pulp can cause ball cushioning and decrease grinding efficiency.
(e) The production of particles smaller than the grind limit *Li* of 0.1 micron involves breakage across the unflawed space lattice, and requires several times as much energy input as the customary breakage along planes of lattice displacement.[6] Considerable amounts of trans-grind-limit particles may be produced in ultra-fine grinding.

When the product size *P* is less than 70 microns the work input *W* as calculated from laboratory tests is multiplied by the following empirical adjustment factor A_1, which equals

$$\frac{P + 10.3}{1.145P}$$

The fine product factor A_1 was derived for the fine dry grinding of cement clinker, and applies to dry grinding down to *P* values of 15 or less. For wet fine grinding, A_1 should have a maximum value of 5.

Conversely, the plant operating work index from Equation (1a) should be divided by A_1 and by $(8/D)^{0.2}$ for direct comparison with the laboratory work index *Wi*.

Proper Grinding Media Sizes

The size of the grinding media is one of the principal factors affecting the efficiency and capacity of tumbling-type grinding mills. It is best determined for any particular installation by lengthy comparative plant tests with carefully kept records. However, a method of calculating the proper sizes, based upon correct theoretical principles and tested by experience, can be very helpful, particularly in new installations.

The general principle of selection should be that the proper size of the make-up grinding media is the size which will just break the largest feed particles. If the media is too large, the number of breaking contacts will be reduced, and the extreme fines made by each contact will be increased. If the media is too small, there will be wasted contacts of a force insufficient to break the particles contacted. In either case the grinding efficiency will be reduced, but the use of undersize media is usually more harmful than the use of oversize media.

Let *B* = make-up ball, rod, or pebble diameter in inches;
F = size in microns 80 per cent of the new feed passes;
Wi = work index at the feed size *F*;
Cs = fraction of mill critical speed;
Sg = specific gravity of material being ground;
D = mill diameter in feet inside liners;
K = an empirical experience constant.

For Ball Mills

In ordinary ball mill operation 1-in. steel balls will effectively grind average siliceous ore with 80 per cent passing 1 mm, or with *F* = 1000 microns or about 16 mesh. It

follows theoretically,[8] and is confirmed by experience, that 2-in. balls are suitable for 4-mm feed, 3-in. balls for 9-mm particles, etc. The ball size should vary as the square root of the particle size to be broken.

From theoretical considerations[3] the proper make-up size B of steel or cast iron balls is found from

$$B = \left(\frac{F}{K}\right)^{\frac{1}{2}} \left(\frac{SgWi}{100\ Cs\ \sqrt{D}}\right)^{\frac{1}{2}} \quad \ldots.(12)$$

The empirical constant K is found by experience to be 350 for wet grinding and 335 for dry grinding.

The commercial size nearest to B is ordinarily selected for the make-up ball size. However, when B is less than 1 in., it may be economical to select a larger ball size for these reasons: (1) the cost per ton of the smaller balls is increased; (2) less wear is obtained from the smaller balls before they are discharged from the mill; (3) the smaller balls may plug the grates of diaphragm discharge mills; (4) large-diameter mills draw more power with large ball sizes.

Ball rationing, which is the regular addition of definite proportions of balls of different sizes, may be used when B is intermediate between two commercial ball sizes, or where an unusual size distribution of the feed requires the addition of some smaller balls with those of the calculated size B.

For Rod Mills

From theoretical considerations[9] the proper diameter B of make-up steel grinding rods is found from:

$$B = \frac{F^{\frac{1}{4}}}{160} \sqrt{\frac{Wi\ Sg}{100\ Cs\ \sqrt{D}}} \quad \ldots.(13)$$

When the reduction ratio $Rr = F/P$ is less than 8, the calculated value of B should be increased by $\frac{1}{2}$ in.

For Pebble and Rockfed Mills

Pebbles fed to pebble mills, and the rock fed to autogenous mills where the large pieces grind the smaller particles, are selected to have the same weight as steel balls suitable for the same service. When B is the proper make-up ball size according to Equation (12), then the proper pebble or grinding rock size of specific gravity Sg is $B \times (7.8/Sg)^{\frac{1}{3}}$.

Size Distribution of Grinding Media

All types of grinding media commonly wear down to sizes sufficiently small to discharge from the mill with the material being ground. However, in some rod mills broken and worn rods are removed manually.

It has been determined that a film of metal of unit thickness is worn from any size ball in a mill in the same grinding time. If the weight loss is periodically replaced as make-up balls of size B, the ball charge reaches an equilibrium size distribution which extends down to almost the ball size discharged from the mill. This equilibrium size distribution follows the equation

$$y = (x/B)^{3.8} \quad \ldots.(14)$$

where y is the percentage of the total equilibrium charge passing any size x. Equation (14) presumably holds for grinding rods and pebbles as well as balls.

In order to obtain consistent performance in wet grinding mills, the initial media charge should be made up from the several sizes available to be similar to the equilibrium charge defined by Equation (14). This can be approximated by drawing on log-log paper the straight per cent passing line with a slope of 3.8 through 100 per cent passing size B. The initial charge composition is determined by marking the points midway between the ball or rod sizes to be used. If ball rationing is to be used, the initial charge should be proportioned between the two ball sizes fed.

In commercial rod mills no rod sizes smaller than $2\frac{1}{2}$ in. should be used in the initial charge. In commercial ball mills the minimum size used is commonly 1 in.

In dry-grinding mills the metal wear rate is so much less than in wet mills that two years or more may be required to reach equilibrium, and the initial charge can be proportioned to fit the mill feed without reference to the equilibrium charge.

The weight, volume, and surface area of steel balls or rods of diameter B inches can be found from Table III.

TABLE III
(B = Diameter in inches)

Number of balls or feet rods	lb.	cu. ft.	Sq. in. surface area
One ball	$0.148B^3$	—	$3.142B^2$
One ft rod	$2.67B^2$	—	$37.8B$
$13{,}500/B^3$ balls	2,000	6.89	$42{,}500/B$
$750/B^2$ ft rods	2,000	5.13	$28{,}400/B$
$1{,}960/B^3$ balls	290	1	$6{,}160/B$
$146/B^2$ ft rods	390	1	$5{,}510/B$
Equilibrium charge fed with B balls	2,000	6.89	$57{,}500/B$

Symbol*	In Equation No.	Description
A	4	Exponential plot slope.
A_1	—	Fine product adjustment factor.
A_2	26	Rod mill Rr adjustment factor
A_3	27	Ball mill Rr adjustment factor.
B	—	Diam. in inches of balls and rods fed.
Bl	5	Blaine surface area in sq. cm/gram.
b	4	Exponential size distribution coefficient.
C	6	Ft-lb per inch, impact crushing strength.
Cl	9, 37, 38, 39, 40, 41	Per cent circulating load/100.
Cp	33, 35, 36	Microns 80 per cent of concentrate passes.
Cr	—	Total crack length—cm/cc.
Cs	12, 13, 19	Fraction of mill critical speed.
Css	11,	Close side crusher setting in inches.
Cw	33	Per cent weight of concentrate in feed.
c	39, 40	Per cent passing in classifier fine product.
cpm	28, 29	Cycles per minute in free vertical oscillation.
D	19, 21–25	Inside mill diameter in feet.
Dp	27, 38	Per cent of discharge passing size P.
d	1	Microns 80 per cent passes.
Ecc	11	Eccentric throw of crusher in inches.
Er	4b	Exponential plot exposure ratio.
F	1, 2, 3, 4, 19, 32	Feed size—microns 80 per cent passes.
Fc	—	Equivalent size of scalped feed.
Fo	30, 31, 32	Maximum efficient feed size in microns.
Fp	38	Per cent of new feed passing size P.
f	5	Diameter of feed particle in microns.
fa	40	Per cent passing in mill new feed.
Gd	33, 34	Grind differential
Gbp	8	Ball mill grindability A–C, net grams/revolution.
Grp	7	Rod mill grindability A–C, net grams/revolution.
Hd	9	Hardgrove grindability
h	28	Inches of free vertical oscillation.
K	—	Grinding media size constant.
Kvo	29	Kilowatts per ton in free vertical oscillation.
Kwb	22	Kilowatts per ton of balls.
Kwr	21	Kilowatts per ton of rods.
k_{100}	2, 3	Schuhmann microns 100 per cent passes.
L	16, 17	Length of mill interior in feet.
Ll	3	Grind limit in microns.
m	39, 40	Per cent passing in mill discharge.
Nc	18	Critical mill speed in rpm.
No	20	Optimum mill speed in rpm.
Oss	10	Open side crusher setting in inches.
P	—	Product size—microns 80 per cent passes.
P_3	13, 14	Sieve opening in microns—A–C grindability.
Q	15	Feet from top of mill to charge.
Qd	23	Feet from top of mill to lowest discharge point.
ρ	3	Density.
Rf	35, 36	Recession factor between concentrate and feed.
Rp	37, 38	Per cent of oversize passing size P.
Rr	4, 5	Reduction ratio $= F/P$.
Rro	25	Optimum rod mill reduction ratio.
Sc	3	Surface area in sq. cm/gram.
Sg	6	Specific gravity of ore.
Ss	24	Ball slump subtraction quantity.
Sw	5	Sq cm per gram, Wagner surface.
Tb	16	Tons of balls in mill.
Tr	17	Tons of rods in mill.
t	39, 40	Per cent passing in classifier coarse product.
Vp	15	Fraction mill volume occupied.
Vpd	23	Fraction of mill volume below discharge level.
W	1, 2, 3, 4	Work input in kWh per short ton.
Wi	1, 6–13	Work index: kWh/ton to 80 per cent—100 microns.
Wt	1, 10	Work total: kWh/ton to product size.
w	—	kWh/Wi
X	4	Exponential plot abscissa.
Y	4	Per cent cumulative retained on any size.
α	2, 3	Slope of size distribution line.
Wi_p	42	Work Index from plot at product size P.
Wi_f	42	Work Index from plot at feed size F.

*In this paper three classes of symbol are used: lower case single letters, upper case single letters and compound symbols consisting of an upper case letter followed by one or more lower case letters. They are distinguished by the sequence rule: lower case single letters are written first, followed by upper case single letters, and in every term the compound symbols are written last.

(*To be continued*)

66.067.374:669.882.883

A SKIMMING PROCESS FOR THE REMOVAL OF OXIDE FROM THE SURFACE OF SODIUM-POTASSIUM ALLOY

Most of the oxides of sodium-potassium alloy (Na-K) float on the surface of the liquid metal at room temperature, therefore it is difficult to entrain the oxides in the flow to a filter. The method used to skim the oxide from the main storage tank of a system handling 1000 gal of Na-K is described in this article. It avoids the use of any moving parts within the Na-K system. A filter which is very suitable for holding a large load of filter cake from the Na-K without blocking is also described

by E. K. INALL

THE homopolar generator being built at the Australian National University[1] will be charged with 1000 gallons of sodium-potassium alloy (Na-K). In operation, the metal at room temperature will circulate through a system of steel pipes into jets which will discharge into a large enclosure filled with pure nitrogen plus 10 per cent helium. Because of the large number of internal partitions, many of which are coated with epoxy resin, the Na-K will come into contact with large surface areas on which there will be adsorbed layers of oxygen, water vapour and other contamination with which it will react when the circulation first starts. The method of removing the products of these reactions, after they have worked their way back into the sump, is the subject of this report.

It is well known[2] that when a metal system is first charged with an alkali metal some tarnishing occurs, even when the cover gas is free of any reactive contaminant. In addition, small amounts of oxide often enter the system through the filling filters. This means that all systems designed to handle alkali metal must be furnished with means of removing oxides and other products of reaction if clean metal is required.[3] After the system has been flushed by the metal, very little, if any, further reaction occurs in a system made from steel, stainless steel or cast iron, with demountable joints sealed with rubber gaskets.

Although the density of all the oxides of sodium and potassium is greater than 2 g/ml and of Na-K is 0.9, at least 90 per cent of the oxide floats on the surface of the metal at room temperature. When the layer of oxide is thin, it forms a strong skin which adheres to the container at the edges. In these circumstances, the metal can be drained from a container as fast as one pleases, but the layer of oxide will cling to the walls of the container. Such a skin of oxide can be broken into a dust by means of a rain of clean metal. The dust floats on what can be a bright metal surface between the spots of dust, but again it will not readily run out if the container is drained. This is the only way in which oxide formed in the many parts of the homopolar generator will get back into the sump. Rafts of oxide more than 2 in. deep float on the surface, presumably because they contain many cells of gas. These also will not flow out when the container is drained through any normal pipeline.

This scum interferes with the operation of level indicators and flow meters and is slowly introduced into the flow, so causing blockages of jets. Therefore, it is important to be able to clean it from the surface of the metal in the sump into which, because of the rain effect in the rest of the circuit, it will tend to migrate.

Conditions for Removal of Scum

There are two features of the method employed to skim the scum from the surface which are essential to its successful operation. First, the strong surface skin is broken by many large bubbles of cover gas rising from below the surface and causing waves which make the scum move some distance from the source of bubbles. It is arranged so that the scum moves towards the skimming outlet (see Fig. 1). Secondly, the skimming outlet is arranged as shown in Fig. 2, to ensure that the metal and the cover gas, which are alternately forced through the outlet, carry with each discharge a section of the metal surface with its scum. This is achieved by sloping the pipe from the skimming outlet so that it falls by at least 1½ diameters before it empties into the separating drum. Thus, when the gas bubbles up through the Na-K in the sump and increases the pressure there, it increases the rate of flow of metal down the skimming pipe until a bubble of gas forms at h_2. The level drops rapidly until the gas escapes into the separating drum in a rush. Because of the momentum of the moving fluids, the pressure of gas drops and the exit of the pipe fills with Na-K again until the next bubble forms.

The action of the waves rapidly dislodges any scum which is adhering to the walls of the vessel within the height of the waves. Thus it is possible to remove scum from the walls of a vessel as the level is lowered, by generating surface waves. The skimming outlet is about half-way down the sump to provide a large surface area on to which most of the scum can be dislodged from the walls before the skimming is started.

The sump tank is 3.5 ft in diameter and 20 ft long. Three

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½ in. diameter gas supply lines are fitted as shown in Fig. 1. The one farthest from the skimming outlet is opened first. When the waves generated by the bubbles and the flow of metal have cleaned the scum from this end, the centre line is opened and the first one closed, so that the scum is driven farther along the surface, until it is suitable to use the last supply point nearest to the outlet. If the sump is full of metal which needs to be cleaned, the sump is vented to the gas holder and the gas supply to the first bubbling point is opened. The skimming outlet is then opened to the separating drum and Na-K is pumped through the filter to a storage tank. When the level has dropped until the surface is level with the centre of the skimming outlet, the delivery from the filter is diverted back to the sump, entering at the end farthest from the skimming outlet. The flow is maintained at between 10 and 30 gallons per minute, depending upon the loading of the filter. The gas vent from the sump is closed and a flow of 6 cu ft per minute of nitrogen is passed through the bubblers so as to induce the skimming action described above.

After the scum has been removed from the surface, the remaining Na-K is pumped through the filter so as to collect any solid contaminants in it. When the clean metal is returned to the sump, the surface remains untarnished until impurities are carried in from some contaminated part of the plant.

With the gas and Na-K flows stated, it took about 10 hr to clear the gross layer of scum which formed when the system was first charged with Na-K. After this time, about 3 lb of solids were skimmed from the surface of the metal in the bottom of the separating drum. Subsequent cleaning of the Na-K has been achieved in less than 3 hr of running.

Two other methods of getting the scum from the sump were considered. In the pilot plant, a movable jet of Na-K was arranged above the metal surface and directed towards the skimming outlet. Although this created a clean area in the immediate vicinity of the impact of the jet with the surface, no general movement of the scum could be achieved. After several unsuccessful attempts to obtain some ejector action, this method was abandoned in favour of the method described above. The other method under consideration was to de-gas the scum with ultrasonic vibrations so that it would sink or go into suspension in the Na-K, from which it could then be collected in the filter. This has not been tried.

Special Design of Filter

A special filter was developed for use in the cleaning circuit. Because of the fine jets used in the homopolar generator, a filter aperture of 0.003 in. was chosen. The solids collected from the circuit rapidly built up on even the largest area of 0.003 in. gauze which it was practicable to use. When the layer was about ¼ in. thick, it represented a formidable impedance to the flow. This was overcome in the filter shown in Fig. 3. The steel wool pad (1) acts as a filter in depth. It is laced to the support (2) by stainless-steel wire. Any small particle of steel wool or solids in the Na-K which leave the pad are collected by the 0.003 in. stainless steel gauze (3). The bulk of the solids is collected in the pad, and when a filter cake of fine particles builds up on the gauze the pad can be drawn along the gauze by means of the rod (4). This compacts the filter cake at one end of the gauze and into the steel wool pad where it is held, leaving the gauze clear to allow a flow of Na-K.

A second reason for adopting this design is that when the filter is loaded with solids and the pad has to be replaced, the liquid metal in the pad can be pressed from it by drawing out the handle until the pad is squashed between the end of the filter and support plate (2), and at the same time the gauze is cleared of any filter cake. In addition to saving the Na-K, this greatly reduces the tendency for the pad to ignite when it is removed from the filter.

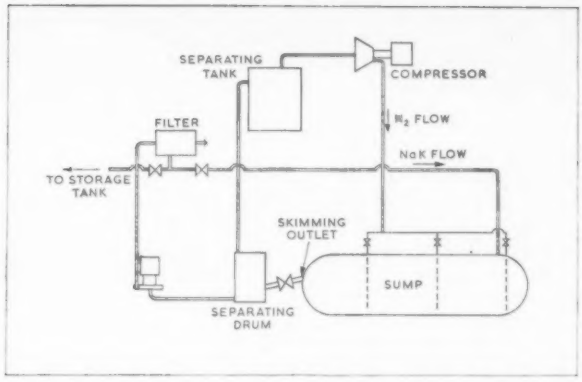

Fig. 1. Arrangement of equipment for removing scum from liquid metal surface; cover gas bubbles form waves which exert a cleaning effect on the molten Na-K alloy in the sump.

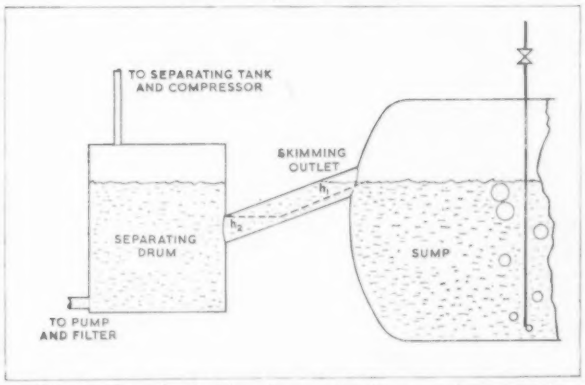

Fig. 2. Detail of skimming outlet for transference of part of metal surface, scum and cover gas from sump to separating drum.

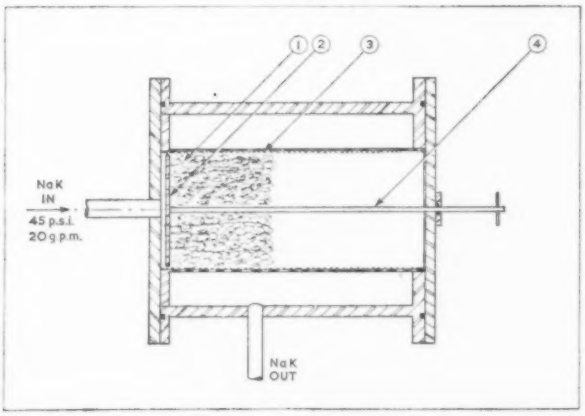

Fig. 3. Cleaning circuit filter comprising steel wool pad (1), support (2), stainless-steel gauze (3) and movable rod for clearing gauze (4).

The equipment was built and tested in the Research School of Physical Sciences of the Australian National University, with the valuable assistance of MR. R. G. HAWKINS, who also contributed many ideas on the design.

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[2] *Liquid Metals Handbook*. Sodium Na-K Supplement, p. 94. November, 1955. U.S. Government Printing Office, Washington 25, D.C.

66.048.3:621.577

THE HEAT PUMP IN MULTI-COMPONENT DISTILLATION

A method of reducing the energy requirement for separating mixtures by distillation is described. By means of techniques proposed, it is shown that the distillation can be carried out at a reflux ratio below the normally accepted minimum value. Practical methods of achieving this are suggested and an example is given showing the possible savings

by D. C. FRESHWATER

ALTHOUGH potentially a reversible process requiring no more work than the net energy difference between the products and the feed, in practice the separation of liquid mixtures by distillation is irreversible and consumes many times the theoretical minimum energy requirement. Since the energy requirement in economic terms is considerable, many methods have been proposed and tried to effect savings.[1, 2, 3] Some of these have had a limited success, others have failed because of the limitations or expense of commercial equipment required such as rotary compressors, very large heat exchangers, and so on. Such methods can be classified into two main groups: those depending upon the multiple-effect evaporation principle and those involving some kind of heat pump, and it is with this latter group that this discussion is concerned.

The heat pump method as proposed in the past depended upon the fact that in many distillation processes the heat to be removed in the condenser at the top of the column is very nearly equal to the heat put in at the bottom by the reboiler. The input is at a higher energy level, however, and so to re-use the heat rejected in the condenser, its energy level (in practice, its temperature) must be raised by some form of heat pump. If this could be achieved, then an approach could be made to operating the separation near the minimum work requirement.

The great disadvantage of this method is that it requires the heat pump to operate over the maximum temperature difference that exists in the system. This is not only thermodynamically less efficient, but imposes practical limitations on the application of the technique.

If the heat pump could be applied over a smaller temperature change it would become a more interesting and technically feasible possibility. It was shown qualitatively by the author that this was possible for binary systems in a paper some years ago, and FRANKLIN and GOULCHER proved that a similar technique could be applied to separate a three-component mixture.

It is proposed in what follows to examine this technique in more detail and to show how it may be of particular value in multi-component fractionation.

Application to Binary Systems

The process will be easier to follow if its application first of all to binary systems is examined. Consider the binary system for which equilibrium data is represented by Fig. 1 and suppose that it is desired to separate a feed at its boiling point (composition x_F) into a distillate, x_D and residue x_W. The operating lines in Fig. 1 show the minimum reflux condition which it is impossible to realise in practice. However, it is seen that the limiting condition applies strictly at a single point. If one could artificially increase the reflux rate at this point, the limiting "pinch" would disappear and the separation would be theoretically practicable with a finite (although very large) number of stages. To make it feasible, consider increasing the reflux ratio over a relatively narrow concentration range, x_1 to x_2. The new condition would now be represented by the equilibrium curve and operating lines of Fig. 2. It is clear that conditions are such that the separation is now quite practicable in a real

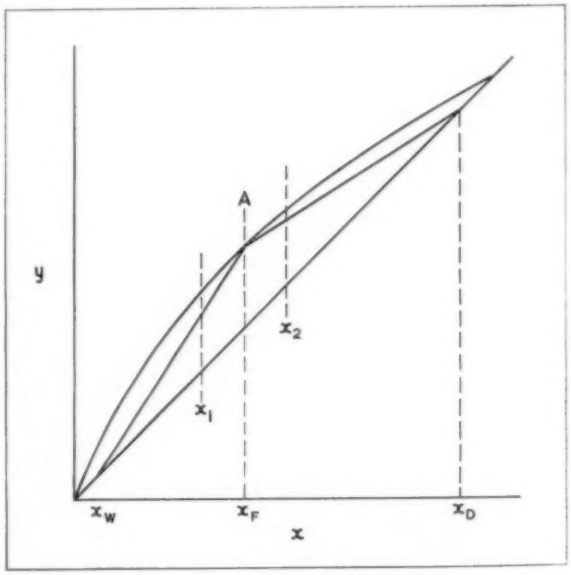

Fig. 1. *Simple "pinch" condition in ideal two-component system.*

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column, although for a very large part of the concentration range the column is operating at the minimum reflux rate.

It can be seen that by extending the concentration range over which one artificially increases the reflux rate one could actually perform the separation with part of the column operating at less than what would normally be regarded as the minimum reflux rate. This condition is shown in Fig. 3, and is mentioned here to emphasise the technique rather than to suggest that such an extreme condition would be desirable in practice.

Of course, it is unlikely that one would be interested in applying complex separation techniques with such an easy-to-separate system as is represented by the equilibrium in the figures so far presented. A more real example of a binary system in which this technique might be of value is one which exhibits a natural pinch in the equilibrium relationship; e.g., the ethyl alcohol/water system.

This is shown diagrammatically and not to scale in Figs. 4 (a) and (b), and operating lines are sketched in to show the technique of an artificially increased reflux ratio to effect the separation at a very low overall reflux rate.

In this case the major pinch is caused by the inflection in the equilibrium curve (point *A* in Fig. 4 (a)). To get past this point and to get back subsequently to a reflux at or near the minimum, one must impose conditions on the unit over a narrow concentration range x_1, x_2, x_3, which result in the distortion of the operating line shown in the enlarged section of the equilibrium diagram in Fig. 4 (b). Again, the fractionation of the mixture over the major part of the concentration change would be carried out at or near the minimum reflux ratio without requiring an unduly large number of stages.

The obvious question that will now be asked is how is this artificial change in reflux rate with the corresponding distortion of the operating lines brought about. Before dealing with this, however, it is proposed to show how the principle outlined above may be applied to multi-component systems.

Application to Multi-component Systems

In considering the application of the technique to multi-component distillation, it is convenient first to consider this on the same basis of graphical construction as that shown for two-component systems. This may be done by using a modified equilibria diagram for the two key components of the type suggested by JENNY and CICALESE.[4] This is shown in Fig. 5 for a typical petroleum light fraction on which are drawn approximate operating lines for a minimum reflux condition. It must be emphasized that this diagram is only approximate, but is useful in showing clearly the pinch points and also in indicating how the technique that is being described may be applied. It is seen by examination of Fig. 5 that by providing additional reflux over a narrow concentration range at each of the pinch zones, the separation over the rest of the concentration range can be carried out at a reflux rate much below that considered normal.

To examine the general case quantitatively is not possible, but an analytical treatment of the problem for three-component systems has been developed by FRANKLIN and GOULCHER,[5] who show that a column may be operated at less than the minimum reflux ratio for most of its sections, provided additional reflux is provided at the important pinch sections.

The Realization of the Technique

We come now to consider how the problem of providing additional reflux at certain specified points in the column may be solved. The problem must now be considered in relation to real apparatus used for fractionation. In practice, this will most commonly be a plate-type column and the subsequent discussion will relate to this kind of equipment. Thus the problem is how to produce a step increase in the reflux rate at a designated plate and to reduce this in a step manner at another designated plate below the first plate. Given suitable heat exchange equipment, this may easily be accomplished by cooling the liquid on the first plate and heating the liquid on the second plate. In small columns this could be achieved with cooling coils, but the technique is of most value in large installations, and the most practical method would be to provide external heat exchangers through which the column liquid is circulated. Now if the cooling is done by a heat exchange fluid rejected to a sink and the heating by another fluid similarly rejected, e.g., cooling water and steam, the saving in total energy achieved by this modification will be so slight as to be negligible for

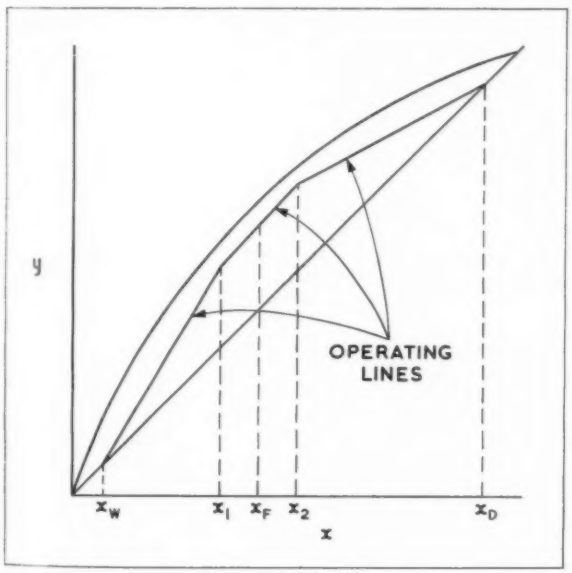


Fig. 2. Avoidance of "pinch" in ideal binary system.

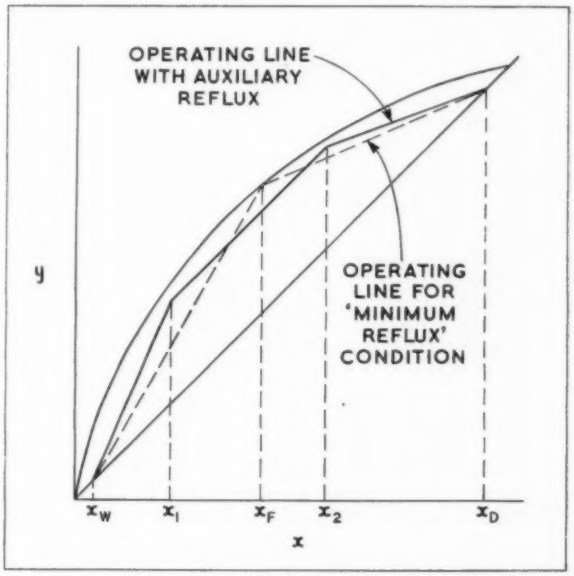


Fig. 3. Extension of auxiliary reflux line to give operation at below minimum reflux conditions.

practical purposes. To take advantage of this technique to make substantial energy savings it is essential that the two heat exchangers be connected via a heat pump. Thus the heat rejected in the cooler is raised in potential to be re-used in the heater. So far this may seem to be little different from the already well-known methods of applying a heat pump across the complete column. There are, however, two important differences which make this technique of much greater potential value. These are, first, the fact that the heat pump is applied across a pinch zone which is by definition a region of small concentration change and hence one over which there is only a small temperature difference. Thus the efficiency and indeed feasibility of the heat pump is greatly improved. Secondly, the column operates over most of its sections at or near the minimum reflux rate and hence considerable reductions in column diameter are possible. It is likely that the resulting economies would be more than sufficient to cover the cost of the additional heat exchange equipment required.

A simple line diagram for a distillation process employing the technique described above and hence a pinch zone in the rectifying section and one in the stripping section is shown in Fig. 6. Interstage heating and cooling by removal of liquid side streams and their passage through external heat exchangers is a technique well known in the petroleum industry and presents no new technical problems. The heat pump unit is thus the only new feature from an application viewpoint, and since a heat pump is simply a refrigerator working in reverse this problem is not in itself novel.

The combination of these features should be realizable without great difficulty.

However, even this complication may not in certain cases be necessary. If one considers a system with a relatively high proportion of heavy components and hence a large mass flow of residues, then it may be possible to operate the heat exchanger system shown in Fig. 7, whereby heat in the residue is used to heat the auxiliary reflux producer *A*. Similarly, the heat in the side stream removed for cooling at *B* is used to provide the energy required for auxiliary reflux at *C*. Clearly, this is a special case and the correct balance of material and energy will only occur infrequently, but it is of potential interest and demonstrates the possible value of this technique.

Quantitative Analysis

Finally, in order to demonstrate quantitatively the potential savings that are possible, an actual example will be considered. This is based on the separation of a petroleum fraction—in fact, a gas stabilization operation. The separation calculations for such an operation have been published by ROBINSON and GILLILAND,[6] and in order to get a direct comparison the same feed product and residue compositions have been assumed. These are summarized in Table I together with other relevant data about the system.

For the present example it was decided to start the separation calculations at the distillate end using a reflux ratio less than the "minimum". The value of reflux ratio used was 0.6, this being chosen arbitrarily. With this reflux ratio, satisfactory separation was found down to plate 7, but from plate 7 to plate 9 the changes in the key components were less than an arbitrarily defined minimum (these were 0.2 mole per cent for the light key and 0.5 mole per cent for the heavy key). It was assumed, therefore, that auxiliary reflux would be required from plate 7 downwards to some subsequent plate. Further calculations were then carried out from plate 7 with a reflux ratio of 2.0. By trial it was found that this additional reflux need only be assumed to apply over plates 7 and 8; thereafter the original reflux ratio of 0.6 could be used as the pinch zone had been passed.

At plate 9 the ratios of key components in the liquid were found to be close enough to that in the feed (0.91 vs. 0.833) to allow this plate to be made the feed plate.

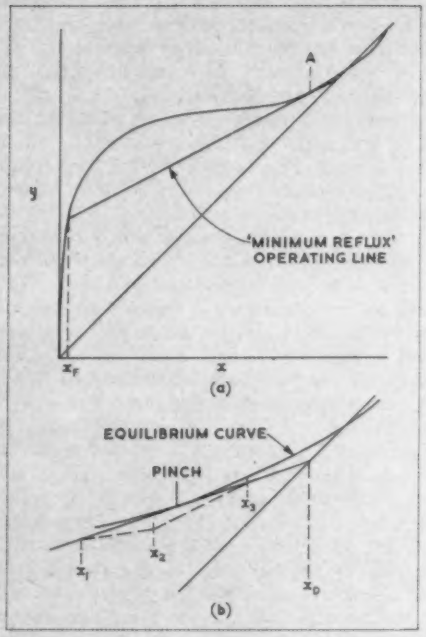
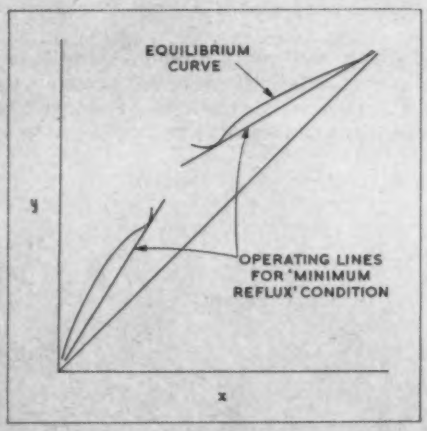


Fig. 4(a). Minimum reflux condition for non-ideal binary system. Fig. 4(b). Enlarged view of pinched zone for non-ideal binary with auxiliary reflux operating lines shown.

Fig. 5. Representation of multi-component mixture on McCabe-Thiele type diagram.



It was assumed that the feed was in the same thermal condition as that in ROBINSON and GILLILAND'S example and thus the new reflux ratio for the stripping section was determined and the separation calculations completed working up from the residue composition. No pinch zone was found in the stripping section under these conditions, and thus the remainder of the separation was carried out at this low reflux rate.

The results of the separation calculations are summarized in Table II.

The next step was to compare the energy requirements

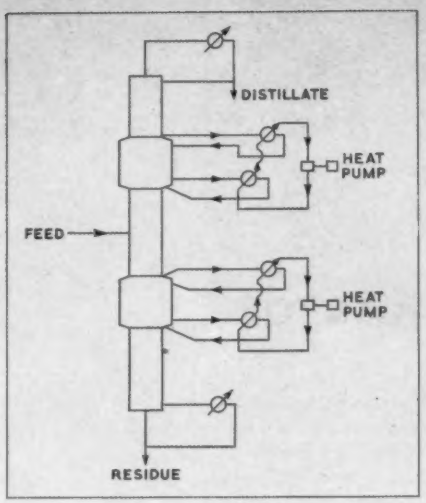
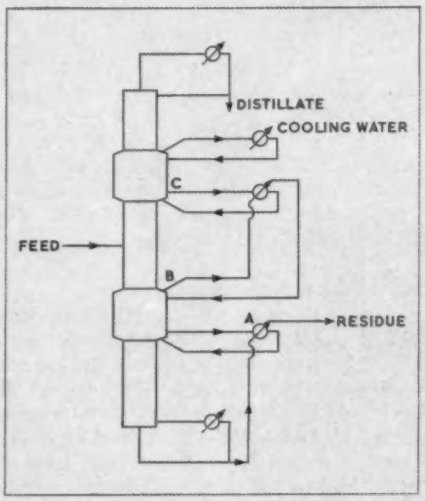


Fig. 6 Simple line diagram of proposed hook-up for providing auxiliary reflux.

Fig. 7. Simple line diagram showing provision of auxiliary reflux in special case without use of heat pump.



of the two methods, and for this purpose it was assumed that a heat pump was available to provide the auxiliary reflux in the manner outlined above. Allowing for temperature drops across the two heat exchangers over which the heat pump operated, it was possible to calculate the total energy requirement of the separation performed in this new way as against that used by conventional methods. These results are summarized in Table III, which shows the saving in this case to be nearly 20 per cent. This is an example taken without thought to show special advantages of this proposed method of operation. Doubtless other separations could be chosen which would show much greater savings. Moreover, it was not possible to perform the calculation for more than one set of rather arbitrary conditions because of the labour involved. Nevertheless, this example does show the possibilities of the method and is sufficiently promising to encourage a more detailed investigation. This is now proceeding, and by using a computer it is hoped to be able to examine many different sets of conditions.

Conclusions

The author suggests that the technique outlined above holds out great promise of effecting substantial economies in the energy required for difficult multi-component distillations. Further theoretical work and calculations of actual examples are necessary and these are in progress. Meanwhile, it is also necessary to examine the practicability of operating a heat pump in conjunction with a distillation column which will involve the solution of a number of interesting problems. Experimental work on anything less than a fairly large pilot-plant scale will be very difficult, because heat losses will tend to mask other heat effects which it is desired to measure.

TABLE I

Component	Feed	Mole per cent Distillate	Residue
CH_4	2.0	6.33	—
C_2H_6	10.0	31.60	—
C_3H_8	6.0	19.00	—
C_3H_8	12.5	39.00	0.25
$i\text{-}C_4H_{10}$	3.5	2.2	4.10
$n\text{-}C_4H_{10}$	15.0	1.9	21.10
C_5H_{12}	15.2	—	22.20
C_6H_{14}	11.3	—	16.50
C_7H_{16}	9.0	—	13.20
C_8H_{18}	8.5	—	12.40
> 360°F	7.0	—	10.24
Operating pressure: 250 psig. Minimum reflux ratio: 0.75.			

TABLE II—Summary of Separation Calculations

	Robinson and Gilliland	Author's Conditions
Plates above feed	7	9
Plates below feed	9	9
Feed plate number	7	9
Reflux ratio	2.0	0.6
Auxiliary reflux ratio	—	2.0
Plates across which auxiliary reflux is applied	—	7 to 8 inclusive

TABLE III—Basis 100 lb. mole of Feed

	Robinson and Gilliland Btu	Author's Conditions Btu
Heat added to system: reboiler	368.2	289
Heat removed from system in condenser	294	156.2
Heat used by heat pump	—	8.9
Difference between heat input in conventional system and new system: 368.2 − 297.9 = 70.3 Btu or 19 per cent.		

ACKNOWLEDGMENTS

The author would like to thank Mr. N. J. Patel who performed most of the separation and heat balance calculations in the example quoted above.

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66.061.4

A NEW TYPE OF SOLID-LIQUID EXTRACTOR

A new type of continuous solid-liquid countercurrent extractor consists of two columns connected by a base of special design. One column provides intimate contact between solvent and dispersed solid; the other, fitted with a helical blade, elevates the solids to the level of their introduction into the extractor. High extraction efficiencies, of 80-95 per cent, and HTU values of from 0.7 to 1.1 ft have been obtained on two liquid-solid systems

by H. L. RAMOS and R. B. ZAYAS

THE main types of solid-liquid continuous extractors may be classified as moving bed, immersion, drained solid and dispersed solid extractor.

Moving Bed Extractors

The Bollman extractor, which first appeared in Germany and was modified in the United States, consists of a series of baskets with perforated bottoms. These baskets are attached to a conveyor which carries them up one side of the extractor and down the other. In their descent they are automatically loaded with the solids and a weak solution is sprayed upon them. The solution passes through the solid bed by gravity and is discharged to the next basket through the holes in its base. The extract, rich in solute, is obtained in the lower part of this part of the extractor. When the basket ascends, the solid is treated with a weaker solution, produced from fresh solvent sprayed into the upper section. The weak solution in this side of the apparatus is collected at the base and pumped to the upper part of the other side; when a basket leaves the top of the extractor it is automatically unloaded, thus completing the cycle.

Two other types of moving-bed leaching machine are the Rotocel[12] and the Miag Wheel.[13]

Immersion-type Extractors

Two solid-liquid extractors are worthy of note; one, devised by HILDEBRANDT,[9, 12, 13, 20] consists of three sections forming a "U", each with a screw conveyor. The solid feed is introduced at one limb, travels downwards slowly by the action of the screw conveyor, enters the middle section and then proceeds upwards in the third. The solvent is introduced in this section and it is countercurrently in contact with the solids.

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The other, developed by BONOTTO,[5, 9, 12, 13, 15] and the subject of many modifications, consists of a series of circular slotted plates arranged one above the other. Each plate has a rake attached to a central shaft; solids are introduced at the top, distributed over the top plate by its rake and pushed through perforations to the next plate below, the solvent flow being countercurrent to the solids.

Drained Solid Extractors

This group is represented by the KENNEDY extractor[17, 20] and the solids in it are leached in a series of tubs arranged in cascade. Solids move up the cascade and are transferred from one tub to the next by paddles; the solvent flows down the cascade and the solids are drained from one tub, or stage, to the next, by perforations in the paddles.

Dispersed Solids Extractors

With this type of extractor the solids are dispersed mainly by agitation in a continuous liquid phase, the solvent. Examples of the dispersed solids extractors are: classifiers, thickeners, agitated tanks with continuous separation of solids, etc. The extractor discussed in this report follows this classification.

In order to increase the rate of flow of a solute from a crushed solid material to a solvent, it is necessary to provide adequate agitation. Generally, in the usual leaching operations, the solids have higher density than the solvents. In an axially agitated vertical column, the solids dispersed in a continuous liquid phase would describe a spinning descending path with a tendency for them to accumulate at the bottom. The solid raffinate could be eliminated by a simple device, such as a screw conveyor, or by directing the raffinate and solution to a centrifugal separator and returning the solution to the bottom of the column; a screw conveyor, however, if vertical, will fail if the liquid phase predominates over the solid phase, or if it has a steep slope.

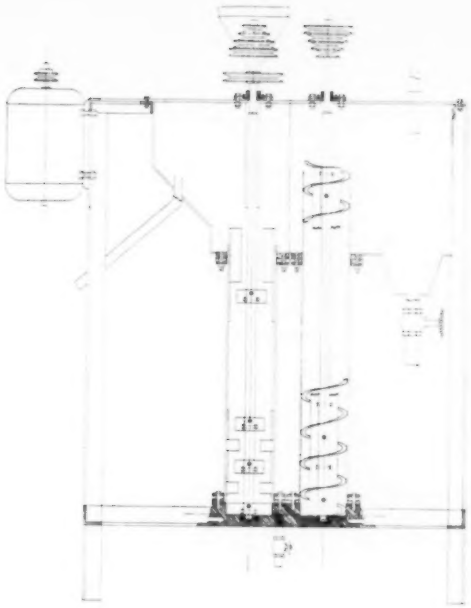


Fig. 1. Solid-liquid extractor.

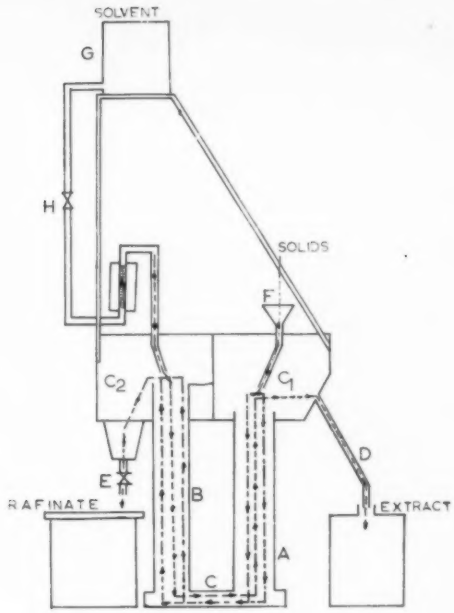


Fig. 2. Flow sheet of Lixiviation process.

it would be larger than the vertical column. The centrifugal separator would increase considerably the cost of the equipment and maintenance of the leaching operation—a solution to these difficulties is offered by the design shown in Fig. 1.

The device used to elevate the solids in this apparatus is a vertical column provided with a long, vertical agitator and a helicoidal metal strip fixed to the wall of the column. When a solid-liquid suspension is agitated axially, the centrifugal force provides a higher relative concentration of solid to liquid and the solids tend to move away from the centre of agitation. Hence, if a helicoidal strip is fixed to the wall of the agitated vessel, it will cause liquid streams to form which fluidise the solid particles. By the proper design of the strip and the action of the agitator, the liquid streams can elevate the solids to the level at which they were introduced into the apparatus.

An extractor of this type has two great advantages. (a) The length of the column may be as large as desired and the problem of transporting solids and solvent would be the same. The length of the columns could be modified by units to be assembled. (b) Single and shipper devices might be used for the separation of raffinate from the solution.

Description of Apparatus

The columns A and B (Fig. 2) were first constructed with Pyrex glass tubes and subsequently of Lucite tubes, 100 mm in diameter. The base and the tanks C_1 and C_2 were made of iron.

Column A. In this column, 20 in. long, were located six turbine-type agitators, constructed from small rectangular strips of mild steel attached to a squared shaft. The shaft speed was 440 rpm, this being provided by a $\frac{1}{3}$-hp 1750-rpm motor through a V-belt drive. The base had a bronze bushing and a steel ball to support the shaft (Fig. 1). Placed between each turbine was a set of three baffles joined together by an m.s. ring. The baffle sets were attached by steel strips of the same length as the column.

Column B. Inside the 24 in. length column was fitted a helix made from m.s. strip; this was made by welding the ends of $\frac{1}{2}$-in. with "O" rings radially cut from m.s. plate. For agitation in this column, four strips of steel plate $1\frac{1}{4}$ in. $\times$ 25 in. were attached to a squared shaft, the latter being supported in the same manner as that of column A. Speed variation was obtained by the use of a set of four V-belt pulleys fitted in the reverse manner of those attached to the shaft of column A.

The base of the columns made of m.s. had two holes 96 mm in diameter and $1\frac{1}{4}$ in. in depth connected with two squared channels $1\frac{1}{4}$ in. by side. In the centre of each hole was fitted a bronze bushing. A cover for the base is shown in Fig. 3b, the holes being 102 mm in diameter to allow the glass tube to enter. The liquid seal between the tubes was made by means of a soft packing material compressed by a circular iron ring.

The lowest turbine on the shaft of column A is larger than the others and is located in the corresponding recess of base C. This turbine causes a stream of solids and solvent to enter tangentially at the bottom of column B through one of the channels of base C, where the solids coming into contact with helix begin to ascend. The solvent is returned to column A through the other channel of base C.

The tops of columns A and B are connected with tanks C_1 and C_2 respectively. At tank C_1 almost all the solids elevated with the extract can be settled and return to the agitation column A. The extract is drained from tank C_1 through a pipe by gravity. At tank C_2 the solids encounter a weak solution due to the addition of fresh solvent in this tank. For laboratory runs, the method chosen for removing the solids from tank C_2 was by opening a valve, located at the bottom of the tank, passing the raffinate and weak solution over a sieve, separating the solids and returning the

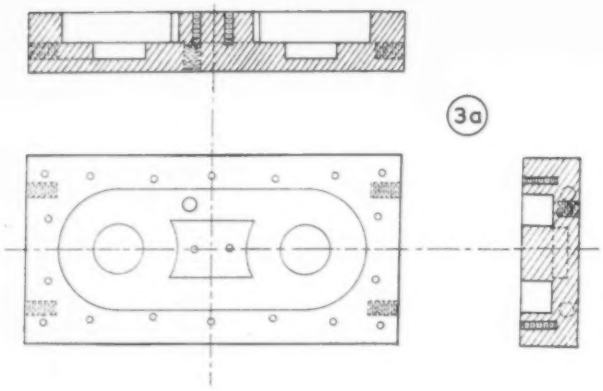


Fig. 3a. Base "C" of the solid-liquid extractor.

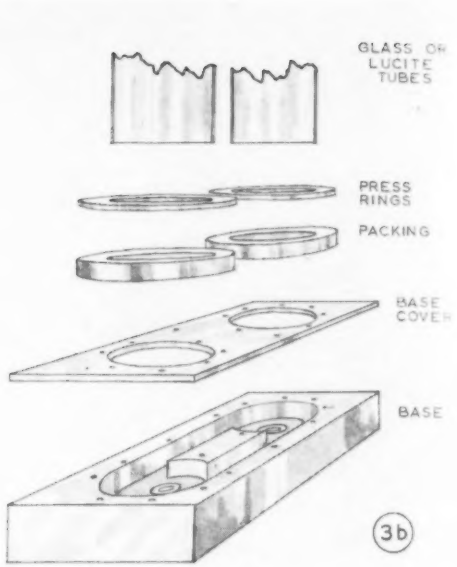


Fig. 3b. Details of installation of base C of the solid-liquid extractor.

Fig. 4. Refraction index v. crude oil concentration in solution.

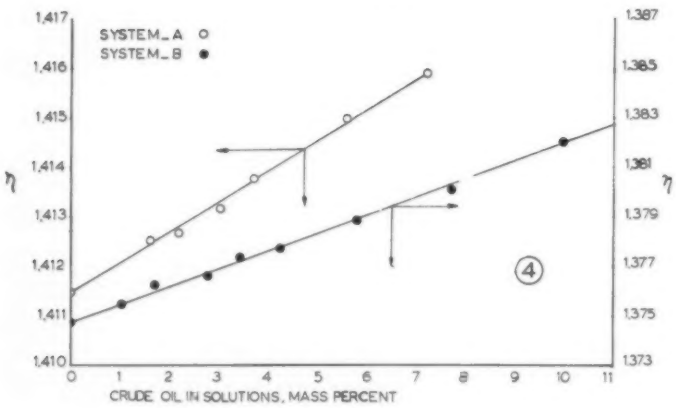
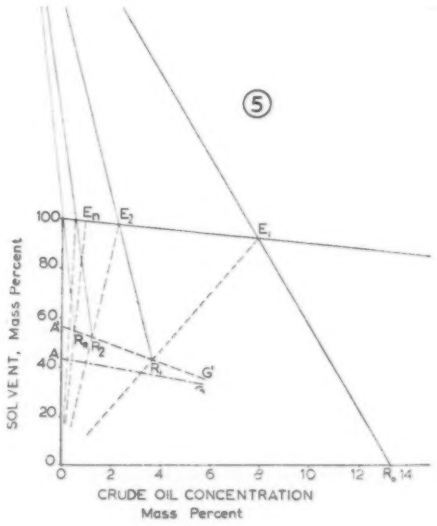


Fig. 5. Theoretical contacts for Run No. 3, System B = 2.77.



weak solution manually to the same tank. For a pilot plant, an inclined screw conveyor could be used for this purpose, with the advantage that fresh solvent can be added to the raffinate in the screw conveyor, a more economical process.

Experimental Work

The experimental study of the extractor was made on two systems: expeller cotton seed cake with naphtha (System A) and press olive seed cake with hexane (System B).

The cotton seed cake was ground to an average particle size of 6 mesh, while the olive seed cake, when dried, formed particles of the same size.

The work was carried out at ambient temperature, the two principal variables studied being: solids rate of flow, F, as grams per minute of dry solids, or S' in lb/hr ft^2, and the weight ratio of fresh solvent to solids feed, R.

A previous study to determine the speed of the shaft in the ascension column in which shaft speeds were 235, 403, 800 and 1350 rpm showed that 800 rpm gave a reasonable transport of solids in the 96-mm-diameter column.

The composition of the solids to be extracted was determined according to the A.O.A.C.[2] Table I shows the average composition of the processed solids.

The time for a run was 2.5-3 hr. The addition of solids was made manually, and a calibrated rotameter was used

TABLE I.—Average Composition of Solid Materials

		Expeller cotton seed cake	*Press olive seed cake*
Wet basis:	Moisture	7.50%	8.40%
	Crude oil	6.41	12.25
	Inerts	86.09	79.35
Dry basis:	Crude oil	6.94%	13.38%
	Inerts	93.06	86.62

for the addition of solvent. Samples of raffinate and extract were obtained at intervals of 15 minutes after the first hour of operation until constancy of solute concentrations was observed. Determination of concentration of crude oil in extract and solution accompanying the raffinate were known from respective refractive index values. Previous determinations of refractive indexes were made for crude oil and solvent for both systems, and these are shown in Fig. 4. The analysis of raffinate was made as follows: a known weight of it was taken to stove at 110°C; the difference in weight corresponded to solvent and water. The solids were extracted in a Soxhlet apparatus, then dried to take out the solvent used—the difference in weight gave crude oil quantity and the remaining solid was considered as inert solid.

Residue Lines

To determine the residue line for a triangular diagram, six single stages with solvent and solid were formed. For this, solvent and solids were introduced at different proportions in Hannus flasks, and after a reasonable interval of time the raffinate and extract were separated in the same manner as would be practised in the laboratory unit. The extract and raffinate were weighed. Fig. 4 was used to determine the concentration of crude oil in the extract and the analysis of raffinate was made in the manner described previously. The results gave the location of line AG for system A and line $A'G'$ for system B in Fig. 5.

Number of Theoretical Stages

After determining concentrations in samples of the extract and raffinate the number of theoretical stages was determined, for each run, in a modified triangular diagram, as recommended in unit operations text-books.[5, 15, 20] Table II shows the number of theoretical stages determined for each run in both systems A and B. Fig. 9 is a plot relating the number of theoretical stages with mass velocity of solids and the weight ratio of solvent to solids. Fig. 5 represents the solution for the number of theoretical stages for run No. 3 for the system B.

Extraction Efficiency

For the determining of the extraction efficiency, samples of raffinate and extract were taken after equilibrium in operation was established. Crude oil was determined in the solution accompanying the raffinate, as well as in the solids, by refractive index observation and an exhaustive extraction in a Soxhlet apparatus respectively. To calculate the efficiency, it was considered that the oil in the solution accompanying the raffinate and the oil in the extract was the total of the crude oil extracted, which was related with the oil present in the solid material before the extraction. Table II and Fig. 6 show the efficiencies obtained in the range of F, or S', and R studied.

Number of Transfer Units and Height of a Transfer Unit

To calculate the number of transfer units for each run, the equilibrium and operation lines were determined, as shown in Fig. 8 for run No. 3, system B. The value "y" is weight fraction of crude oil in the extract and the "x" values those

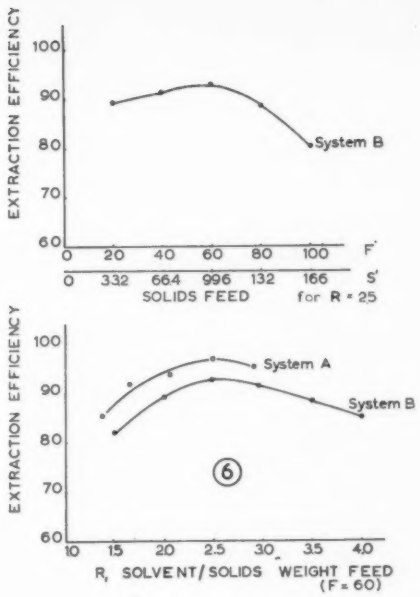
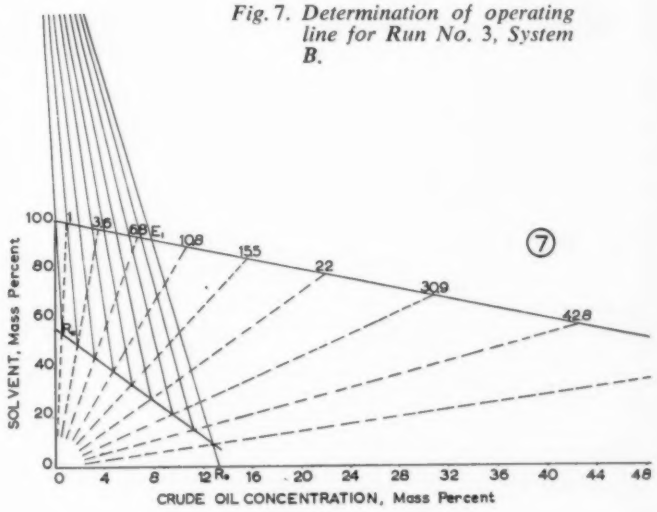


Fig. 6. Extraction efficiency results.

Fig. 7. Determination of operating line for Run No. 3, System B.



corresponding to the solution retained by the raffinate, but free of solids. As, at equilibrium, the concentration "y" is the same as "x", the slope of the equilibrium line is 1. The operation line is determined as follows: from the "difference point" (not shown in Fig. 7) a set of lines are drawn. The limiting lines of this set are those passing over the points corresponding to fresh solvent concentration and composition of the raffinate and compositions of the extract and solids feed. Intersections of the lines with 0 per cent of inert solids axes give the values for "y". The point of intersection of the line from the difference point and line of residues joined with that one for 100 per cent inert solids describe a straight line, which, extended up to 0 per

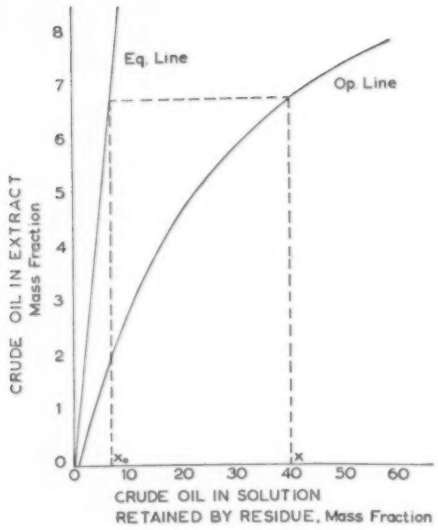


Fig. 8. Equilibrium and operating lines for Run No. 3, System B.

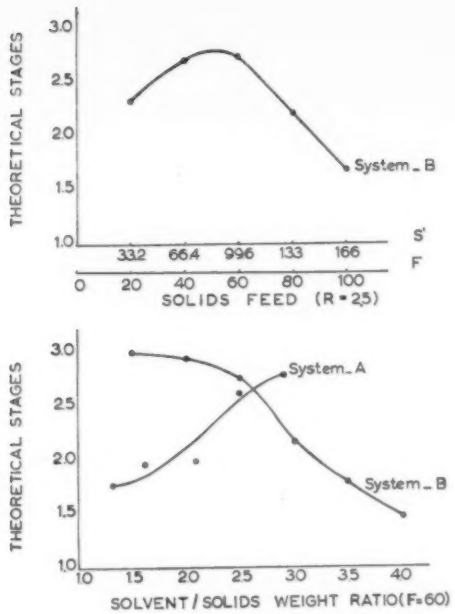


Fig. 9. Number of theoretical stages for the two systems investigated for different solid feed rates and solvent/solids ratios.

cent inert solids, describe the corresponding values of "x". Values of x and y for each line can be determined to locate the operating line. For run No. 3, system B, the following values were obtained:

x	59.0%	42.8	30.9	22.0	15.5	10.8	6.8	3.6	1.0
y	7.9	7.0	6.0	5.0	4.0	3.0	2.0	1.0	0.0

Fig. 8 shows the operating and equilibrium line.

The number of transfer units were evaluated by the calculation of the integral $dx/(x - x^*)$ between the limits $x_1 = 59$ per cent and $x_n = 1.0$ per cent; $(x - x^*)$ is the concentration driving force for diffusion of the solute from the solution near the solid to the whole mass of solution. Table III presents the evaluation for NTUs for this particular case.

Values for the height of the transfer unit (HTU) were calculated from

$$\text{HTU} = Z/NTU.$$

where Z is the equivalent length of the apparatus and equal to 4 ft (height of column A = 20 in.; height of column B = 24 in.; average length of channels connecting the columns = 4 in.). For run No. 3, system B, the value for HTU found was 0.735 ft. Fig. 10 is a plot of both NTUs and HTUs values versus the solids mass velocities, S', or solids feed, F.

Results and Conclusions

(1) For a column diameter of 4 in. and a ratio of diameter of agitator to column of 0.31, a satisfactory ascension of solids was observed when the speed of the agitator was 800 rpm.

(2) It was found a maximum in the efficiency of extraction of 93 per cent and 97 per cent when the solids mass velocity was 95 lb/hr ft² and a weight ratio of solvent to solids feed of 2.5. The efficiency here reported takes account of the crude oil in the solution accompanying the raffinate.

(3) The proportion of solids in the extract was of the order of 1 to 1.5 per cent weight for system A, while for system B it was from 0.5 to 0.8 per cent.

TABLE II.—Number of Theoretical Stages and Efficiency of Extraction for Systems A and B. Number of Transfer Units and Height of a Transfer Unit for System B

SYSTEM A						SYSTEM B						
Run No.	F	S'	R	Stages	Effic. %	F	S'	R	Stages	Effic. %	NTU	HTU
1	60	99.6	2.5	2.63	97.0	20	33.2	2.5	2.33	89.2	5.051	0.792
2	60	99.6	2.08	1.97	94.0	40	66.4	2.5	2.70	91.7	5.751	0.696
3	60	99.6	1.67	1.97	92.0	60	99.6	2.5	2.76	92.8	5.443	0.735
4	60	99.6	1.33	1.79	85.5	80	132.8	2.5	2.21	88.6	4.834	0.828
5	60	99.6	2.91	2.80	95.8	100	166.0	2.5	1.68	80.1	3.653	1.096
6	20	33.2	5.00	1.85	94.6	60	99.6	1.5	3.00	82.3	5.031	0.796
7	40	66.4	2.50	1.86	94.5	60	99.6	2.0	2.95	89.5	5.306	0.754
8	80	132.8	1.25	1.82	88.0	60	99.6	3.0	2.17	91.2	5.425	0.737
9	100	166.0	1.00	1.76	87.0	60	99.6	3.5	1.79	88.5	4.462	0.896
						60	99.6	4.0	1.48	85.1	3.873	1.043

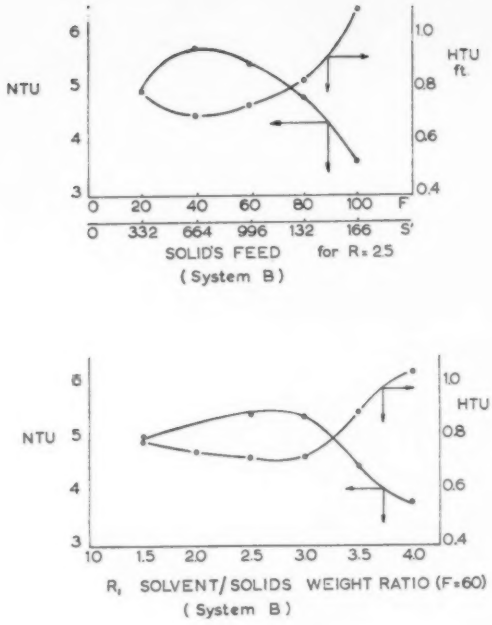


Fig. 10. Estimation of number of transfer units, including height of transfer units.

(4) The solids mass velocity at flooding was of the order of 250 lb/hr ft². Flooding was observed for solids before channels of base of columns.

(5) If extrapolation of line of residues *A'G'* (Fig. 7) is accepted, the maximum number of transfer units found was 5.75 when the solids mass velocity was in the order of 66.4 lb/hr ft², but if the conditions represented by Fig. 10 are observed, there was not a well-defined maximum number of transfer units when the ratio *R* varied, with *S'* constant at 99.6 lb/hr ft².

TABLE III. — Determination of Number of Transfer Units for Run No. 3, System B

x	x^*	$x - x^*$	$1/(x - x^*)$	$x/(x - x^*)$
59	7.90	51.1	0.01956	0.1938
50	7.45	42.55	0.0235	0.1248
45	7.15	37.85	0.02644	0.1428
40	6.80	33.20	0.0307	0.1642
35	6.40	28.60	0.0350	0.1913
30	5.90	24.10	0.0415	0.2310
25	5.36	19.64	0.0509	0.2575
20.5	4.77	15.73	0.0635	0.4118
15	3.87	11.13	0.0898	0.5725
10	2.82	7.18	0.1392	0.5124
7	2.06	4.94	0.2202	0.8301
4	1.15	2.85	0.3510	0.9840
2	0.42	1.58	0.6330	0.8165
1	0.00	1.00	1.0000	
			$NTU =$	5.4327

(6) A maximum number of theoretical stages for system B was observed when *S'* was of the order of 95 lb/hr ft², while an increase of the ratio *R* corresponded to a decrease in the number of theoretical stages. If system A is considered, it gave an increase in the number of theoretical stages when the ratio *R* increased at a constant value of *S'*, or *F*, but the curve seems to descend for higher values of *R*. As temperature and conditions of operation were the same for both systems, it is concluded that the difference in curves in Fig. 9 is due solely to the nature of the solids and solvent used in each system.

Intermediate Pressure Ammonia Oxidation Plant

THE new nitric acid plant at the I.C.I. Nobel Division works at Ardeer, Ayrshire, has a capacity of 55,000 tpa (100 per cent HNO_3) and it replaces three older units which had a total capacity of 40,000 tpa. The new plant produces 60 per cent HNO_3, which is mainly used in nitration processes.

The ammonia required for the oxidation stage is conveyed to the site in 12-ton rail tankers and is stored in 150 ton capacity spherical holders at 0°C, 45 psig. This temperature is maintained by a refrigerating system which recompresses the flashed-off gas.

Before mixing with the air stream, the vaporized ammonia is passed through glass-wool filters. Process air is washed with water in a Peabody scrubber and mixed with ammonia to give an NH_3 concentration of 11.5 per cent. The air and ammonia streams are each preheated to 50-200°C before mixing. A system of ceramic candle filters cleans the gas mixture further before it passes to the convertors. A number of multi-nozzle mixers ensure an homogeneous gas mixture, the composition of which is continuously recorded by a catherometer instrument. The ammonia : air ratio controlling system is controlled automatically and is closely related to the catalyst gauze temperature; the latter is continuously measured and recorded by means of a radiation pyrometer.

The oxidation of ammonia is carried out in three parallel convertors, each employing three rhodium-platinum alloy gauzes 114 in. in diameter. The reaction pressure is 1-2 in. w.g. below atmospheric pressure and the temperature is in the vicinity of 850°C. This is maintained by careful control of the flow rates, preheat temperature and NH_3 : air ratio.

The catalyst is woven from 0.06 mm diameter Rh-Pt wire and forms pads which are clamped into position in the reactor. The loading is 140 lb NH_3 per day per ounce Troy of Pt alloy exposed.

The conversion efficiency is greater than 96 per cent and the overall plant efficiency exceeds 94 per cent. Catalyst alloy losses are expected to be very low, less than 50 m.g. per ton nitric acid, this order of loss being one of the main advantages of the low-pressure oxidation system.

Below each convertor forced-circulation boilers with superheaters and economizers are placed and provide steam to the tune of 15,000-20,000 lb/hr at 300°C and 270 psig. This quantity provides two-thirds of the power required by the turbo compressor which compresses the reaction products in readiness for absorption.

The nitric oxide is oxidized and absorbed in a series of six ring-packed stainless-steel towers, each 60 ft high. The efficiency of absorption can be gauged from the stack loss, which amounts to less than two grains of acidity per cu ft.

Based on an article by J. G. Sloan and H. C. Staats, *Platinum Metals Review*, 1961, **5**, 2, 54.

66.045.1

HEAT-TRANSFER COEFFICIENTS IN CHEMICAL PLANT HEAT EXCHANGERS

A heat exchanger with fouled external surfaces seen at B.P.'s Kent refinery. (Courtesy B.P. Co. Ltd.)

Overall heat-transfer coefficients, measured during the operation under works conditions of examples of each of four common simple types of heat exchanger used in chemical plant, are recorded. Using standard design procedure, overall coefficients have been calculated for common operation conditions. A comparison is made between calculated and measured coefficients. Numerical deviations for averaged results, between the calculated and measured coefficients for individual types of exchanger, working under turbulent and streamline flow conditions, are quoted

by F. R. WHITT

Introduction

THE accuracy with which an overall heat-transfer coefficient can be calculated, using accepted design procedure, compared with the value achieved under works conditions depends upon three main factors. These are the individual accuracies of correlation of relevant data in connection with the estimation of film coefficients, fouling of the heat transfer surfaces and numerical values for the relevant physical properties of the fluids concerned.

It is probable that a peak of accuracy of correlation of experimental data on liquid film coefficients has been reached with the turbulent flow of fluids in single tubes.[1, 2] A maximum variation, either side of a mean value, of ± 20 per cent is claimed. Experimental data for more complicated heat-transfer apparatus of the multitubular type[3] have not been correlated so successfully. The relevant chapters of standard works[1, 4, 5, 10, 13, 18] show that correlations with variations either side of a mean of two- or more-fold the ± 20 per cent quoted above are commonly accepted as the best possible.

The collection and interpretation for design purposes of experimental data concerning the effect of unclean surfaces on heat transmission is a controversial subject.[19] Although, maybe, not so important to the furtherance of heat-transfer practice as are film coefficients, and therefore of less academic interest, the effect of unclean surfaces in industrial exchangers is most important. The relative magnitude of the effect of dirt films, under some circumstances, compared with other factors, on heat transmission is well illustrated by SMITH[20] and BREIDENBACH *et al.*[22]

As a consequence of the type of experience described above, recognized industrial plant design procedure for estimating the effect of unclean surfaces has been established. Allowances, for design purposes, are made in two ways. One procedure uses the concept of fouling coefficients or factors.[6, 11, 14] The other procedure uses a cleanliness factor associated with the value of the clean overall coefficient.[12, 15]

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Data on some appropriate physical properties of the fluids involved are necessary for any work concerning heat-transfer coefficients. These data, for cases in which well-known fluids are involved, are probably known to a greater degree of accuracy than any correlation of heat-transfer coefficients. In the case of less common fluids, however, particularly if estimations of physical properties, such as thermal conductivity values, are made to avoid laborious experimental work,[23] some significant errors may be introduced thereby into heat-transmission calculations.

In view of the above-mentioned sources of possible errors in heat-transfer estimations, it was thought worth while examining particular available data to see how, in practice, the errors combined and to what extent the estimations differed from the practical values. It was also hoped that it would be possible to obtain information concerning the fouling factors, appropriate to certain particular circumstances, for silver and stainless-steel surfaces.

It was assumed that the equipment had been in use long enough for the fouling of the surfaces to have become reasonably constant. This was considered feasible because small fouling only was expected. A discussion is given by KERN[16] on the time factor inherent in the definition of fouling factors.

The heat exchange processes discussed were, in general, those for which reliable correlations of data were given in the literature; i.e., forced convection turbulent flow processes for heating or cooling. The experimental data, reported upon in this paper, supported the reliability of these correlations for calculating film heat-transfer coefficients. From the work done, however, with fluids in streamline flow in tubes, when free convection effects become apparent, the agreement of literature and experimental data was not so satisfactory.

Experimental

(*a*) *Equipment and Fluids Used*

Brief descriptions are given in Table I of all the examples of four main types of equipment used. The source of heat

Symbols Used

U = overall heat-transfer coefficient;
h = film heat-transfer coefficient;
q = heat transferred, C.H.U./hr;
$\triangle t$ = temperature difference, °C;
L = thickness of heat-transfer wall, ft;
k = thermal conductivity of wall material C.H.U./(hr) (sq ft) (°C per ft);
A = area of heat-transfer surface, sq ft;
$C.H.U.$ = heat required to raise 1 lb of water 1°C;
j = a product of dimensionless terms (see references in Table I);
Reynolds Number = length × mass flow rate/viscosity (see references in Table I);
Prandtl Number = specific heat × viscosity/thermal conductivity (see references in Table I);
Fouling Factor = $10^3/h_f$.

Subscripts

EXP = experimental value derived from work described;
$CALC$ = calculated value derived from use of data in the literature;
c = controlling film;
s = service fluid film;
f = fouling film (c or s).

All heat-transfer coefficients in C.H.U./(hr) (sq ft) (°C) units (= Btu/(hr) (sq ft) (°F)).

for the tubular heaters was either circulating hot water, at about 70°C, or low-pressure steam; this latter was also used in the jacket of one reaction vessel. In the cases of the other reactor vessels the dimethyl hydrogen phosphite was heated by electrical immersion heaters. The methylene chloride, originally at about 15°C, was cooled to lower temperatures by the calcium chloride brine served cooling coil in that particular vessel. The warmed carbon tetrachloride from the coils of one reaction vessel was passed through the shell side of the tubes in the shell exchanger. This latter was cooled by calcium chloride brine, at about −15°C, circulating through the tubes.

The liquids used had viscosities of 0.4 to 5.0 centipoises and Prandtl Numbers of 2.9 to 25 were involved. Most of the physical properties data were obtained from the general literature, but some experimental determinations were necessary.

The liquids used were free from suspended matter and of a general commercial standard of purity. The dimethylhydrogen phosphite flowing through the tubular heaters contained, however, some free hydrogen chloride (hence the choice of "0" for a fouling factor—see Table II, column 6).

(b) Correlation of Experimental Results

The experimental overall heat-transfer coefficients, as listed in Table II, were calculated from plant operation data by the use of the equation

$$U_{EXP} = q/(A)(\triangle t) \quad \ldots . (1)$$

The calculated overall heat transfer coefficients were derived from the simplified approximate equation

$$U_{CALC} = \frac{1}{\frac{1}{h_c} + \frac{1}{h_{fc}} + \frac{L}{K} + \frac{1}{h_{fs}} + \frac{1}{h_s}} \quad \ldots . (2)$$

The area of heat-transfer surface used for the calculations was the one associated with the controlling film resistance. This latter was, in general, predominant to such a

TABLE I—Details of Equipment Dimensions and Data Correlation Plots

*Incorporates two fouling film effects. †Fluid associated with the controlling film resistance. ‡Velocity in shell of CCl_4 (see KERN,[13] p. 138).

No.	Type of Heat Exchanger	Details of Tube or Vessel				Details of Stirrer		No. of Exp. Runs	† Fluid Velocity in Tube Range of f.p.s.	Data Correlation Plot Used and Representative Results				References to Plots Used
		Capacity (gal)	Metal	Height or length (ft)	Dia. (ft) or Bore (in)	Width (ft)	Revs. per Minute Range			Ordinate $(j)_1$ From Ref. Plot	Abscissa (Reynolds Number)	Ordinate $(j)_1$ From Exp. Data	Ratio $\frac{(j)_1}{(j)_2}$	
1	Tube with jacket (vertical)		Silver	7	$\frac{1}{2}$			10	3.64 to 4.88	147	50,300	189	0.777	KERN,[13] Fig. 24
2	" " "		"	"	"			8	0.95 to 3.4	67.5	19,320	45.5	1.48	"
3	Vessel with coil	1.1	Copper	0.5	0.5	0.165 (Propeller)	220 to 340	5		275	9,330	192	1.43	KERN,[13] Fig. 20.2, and CHILTON *et al.*[11]
4	" " "	6.0	Silver	1.5	0.83	0.265 (Propeller)	365 to 500	4		720	42,100	386	1.86	
5	" " "	52.0	Nickel	2.3	2.2	1.16 (Anchor)	52	6		2230	247,500	2050	1.08	"
6	" " jacket	25.0	Stainless steel	1.5	1.87	1.75 (Anchor)	100	6		2100	433,000	676	3.16	"
7	Tubes (60) in shell		Stainless steel	2.25	$\frac{3}{8}$			3	0.73 to 0.87	369	305	27.6	1.33	McADAMS,[5] Fig. 83
8	Tubes (52) in shell (no baffles)		Mild steel	3.5	$\frac{3}{8}$			9	‡ 0.0052 to 0.011	3.8	825	4.95	0.767	McADAMS,[5] Fig. 115 (0.6 factor)
9	Tube with jacket (horizontal)		Silver	12.67	$\frac{1}{2}$			12	0.065 to 0.394	9.8	1,555	18.7	0.546	KERN,[13] p. 206, Equation 10.4
10	" " " "		Mild steel	1.485	$\frac{3}{8}$			12	0.304 to 2.19	23	1,755	42.8	0.546	"
11	" " " "			2.89	$\frac{3}{8}$			12	0.325 to 1.78	12	193	20.0	0.54	"

TABLE II—Comparison of Calculated with Experimentally Determined Overall Heat-transfer Coefficients

(Heat-transfer coefficients in C.H.U./(hr) (sq ft) (°C). Col (2) incorporates two unknown fouling film effects.) (Film coefficients for steam in Col. (3) taken as 1,000. Service film coefficients calculated from McAdams's p. 183, Equation 9c, and pp. 167 and 168.)

No.	Type of Heat Exchanger	Heat Transfer Surface (sq ft)	Fluids Inside		Flux Range (C.H.U./(hr) (sq ft))	Δt range (°C)	Controlling Film Heat-transfer Coefficients		Service Fluid Film Heat-transfer Coeffs. (+ Wall) (3) Calculated	Overall Heat-transfer Coeffs.		$\frac{U_{CALC}}{U_{EXP}}$	(6) Fouling Factors used to Calculate (4) (see Ref. 2 p. 264, and Ref. 5, p. 128)
			Tube or Vessel	Jacket or Coil			(1) Calculated	(2) Experimental		(4) from 1, 3 and 6 U_{CALC}	(5) U_{EXP}		
1	Tube with jacket (vertical)	0.98	Methylene chloride	Hot water	13,900 to 9,650	47.16 to 39.13	500	644	522	202	288	0.70	0+1 =1
2	" " " "	"	"	"	5,760 to 2,360	41.6 to 21.35	242	164	239	108	93	1.16	0+1 =1
3	Vessel with coil	1.12	Dimethyl hydrogen phosphite	Cold water	1,560 to 750	16.9 to 9.0	102	71.3	1,285	79	68	1.16	1+1 =2
4	" " "	6.3	"	Cold CCl_4	2,800 to 1,740	22.9 to 20.5	254	136	290	106	92.8	1.13	1+1 =2
5	" " "	32.0	Methylene chloride	Cold $CaCl_2$ sol	1,080 to 150	12.5 to 0.7	152	141	525	115	107	1.07	1+1 =2
6	" " jacket	12.0	Water	Steam	11,800	102.6 to 60.0	741	234	491	185	138	1.34	1+1 =2
7	Tubes in shell (horizontal)	19.0	Water	Steam	8,400 to 5,000	52.0 to 48.5	230	172	730	132	139	0.95	1+1 =2
8	Tubes in shell (horizontal)	30.0	CCl_4	Cold $CaCl_2$ sol	4,850 to 2,500	31.6 to 29.3	11.7	15.3	90	10.1	13.1	0.77	1+1 =2
9	Tube with jacket (horizontal)	0.28	Methylene chloride	Hot water	1,822 to 259	37.0 to 15.0	35	67	250	29.9	52.7	0.567	1+1 =2
10	" " " "	0.24	$CaCl_2$ sol	Steam	23,820 to 14,470	109.0 to 102.0	170	330	500	107	199	0.51	1+1 =2
11	" " " "	0.47	"	"	14,250 to 12,870	87.3 to 10.0	150	210	500	92.3	145	0.61	1+1 =2

degree that the use of the simplified procedures appeared justified.

The experimental controlling film coefficients of Table I were obtained from the measured overall coefficients by subtracting from them the service fluid film coefficient and wall effect. The experimental film coefficients thus embody the effects of two fouling films, one associated with the processed fluid and the other with the service fluid. It was found, nevertheless, that correlation of data could be achieved, in as satisfactory a manner as recorded data in the literature concerning the same process, by plotting j factors versus Reynolds Numbers. The precise composition of these latter two numerical concepts varied, of course, according to the type of process involved. The references quoted in Table I give the definitions of the quantities.

It was found that the most appropriate correlation line, through the experimental data points for controlling film coefficients, was in all cases one which was parallel to the one given in the literature for the particular plot of j factor versus Reynolds Number. The experimental line, however, was, in the case of processes involving turbulent flow, generally lower in ordinate value for a given value of abscissa or Reynolds Number. The respective displacements are given as j_1/j_2 ratios in Table II. It thus appeared that the effect of unclean surfaces could be expressed as a constant factor times the calculated value for the controlling film coefficient. This mode of expression resembles that given in the literature[12] as a recognized procedure for expressing fouling effects in connection with steam condensers. The fouling effect is there quoted as a percentage of the overall coefficient which is controlled by the predominant film coefficient associated with the water side flow.

Table II lists typical point values from the correlation lines on the plots of experimentally deduced controlling film heat-transfer coefficients. The experimental overall heat-transfer coefficients which formed the bases for the particular typical points are also listed. Approved methods for calculating the service fluid film coefficients were used.[7] In the case of the flow of water in an annulus, Nos. 1 and 2 of Table II, the calculated coefficient was found to check with the results deduced from a Wilson type plot.[8] The calculated controlling film coefficients were deduced from the plots described in the various references given in column 6 of Table II.

Most of the experimental work was carried out with the fluids flowing in the turbulent region and the experimental results suggested that the recognized equations for liquid film coefficients were quite reliable. Some work carried out in the streamline flow region (see results 9, 10 and 11 of Table II) suggested that the accepted equation[17] would be liable to give low values for film coefficients in some circumstances. This equation[17] is based upon the correlation of both heating and cooling data, and the experimental results which conflicted with the predictions of this equation were for heating data only. This fact might be a contributory cause of the discrepancy. It appears that the compensation for free convection effects is difficult to assess numerically.[9, 24]

Conclusions

The comparison of calculated overall heat-transfer coefficients, for examples of four common types of chemical plant heat exchanger for liquid/liquid (or vapour) heat exchange, with experimentally determined coefficients shows that:

(1) With turbulent flow conditions the averaged ratios of calculated to experimental coefficients vary from 0.77 to 1.18 (see Table III). This deviation from a mean seems reasonable, because it is consistent with the probable effect of the individual deviations from a mean of the various data used for calculation: *i.e.*

TABLE III—Summary of Results

Nos.	Type of Exchanger	Heat Exchange Process	Flow	Ratio of HT Coeffs. i.e. Calculated / Experimental		Conclusions
				Limits	Average	
1, 2 and 7	Tubes in jacket or shell	Liquid/liquid or vapour	Turbulent	0.7 to 1.16	0.94	Maximum errors −23% +18%
3, 4, 5 and 6	Vessels with coils or jacket	" "	"	1.07 to 1.34	1.18	
8	Tubes in shell	" (crossflow) "	"		0.77	
9, 10 and 11	Tubes in jacket	Liquid/liquid or vapour	Streamline	0.51 to 0.61	0.56	

that of film coefficient correlations and fouling factors.

(2) With streamline flow conditions in short tubular exchangers the ratio averaged 0.56. This means that a recognized method[17] for estimating film heat-transfer coefficients could provide calculated overall coefficients too low by as much as 44 per cent in some conditions.

ACKNOWLEDGMENTS

A considerable amount of the data quoted in this paper was derived from work carried out in the course of plant operation by G. A. HUNT, K. S. GREGORY, E. A. MATTHEWS and A. MCKENNA at the now closed Ministry of Supply Research Establishment, Sutton Oak, St. Helens, Lancs.

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Temperature Control of Exothermal Reactions

The Institution of Chemical Engineers' North-western Branch Meeting

A MEETING of the Institution of Chemical Engineers, North-western Branch, was held at Manchester on March 28, when a paper, "Temperature Control of Exothermal Reactions Entailing Special Heat-transfer Problems", was presented by MR. B. STREET.

The author discussed the temperature control of oxidation and polymerization processes, particularly those in which ethylene participates. The fluidized bed technique is used for processes such as the oxidation of naphthalene. For the oxidation of ethylene by air or by oxygen, a fixed bed catalyst is employed to direct the oxidation to ethylene oxide rather than to complete combustion. The former reaction proceeds at a lower temperature and it is less exothermic than the complete combustion; the heat of reaction must be removed from the catalyst to ensure that the temperature is kept at the optimum for a maximum reaction rate and for the desired degree of oxidation. Resistance to heat transfer on the coolant side of reactors usually is small compared with the resistance on the process side, and the larger the scale of production the more severe is the heat-transfer problem. Another problem is the danger of "hot spots" on catalyst tubes, and to minimize this there must be good heat transfer on the coolant side supplemented by such techniques as staged reaction temperatures, stage dilution of the catalyst and dilution of the reactants with inert gases. The heat liberated at the surface of the catalyst must pass through the mass of catalyst and through the tube wall to its surface, from where it may be removed by the evaporation of a boiling liquid. The temperature of the boiling coolant is regulated by varying the pressure under which it boils, a temperature controller being employed for this purpose. Nucleate boiling should be induced throughout the length of the reactor, with condensation of the vapour and its return as a liquid to the bulk of the coolant.

Heat removal from bulk polymerization processes is also something of a problem, for it is hindered by the high viscosity of the products. Mechanical stirrers assist the strict control of process temperatures required for good quality of product and for its minimum of depolymerization; however, significant temperature gradients through the bulk cannot be prevented and they cause the formation of polymers with a wide range of molecular weights. Polymerization in solution offers a means of reducing overheating of the polymer, but a disadvantage is the slower rate of the polymerization compared with that of bulk polymerization at the same temperature. Some solvents dissolve the monomer, but leave the polymer insoluble. Emulsions of the monomer in water may improve the rate of polymerization, but difficulties of removal of emulsifiers, catalysts and stabilizers from the product arise. Pearl polymers are produced from an agitated suspension of monomer in a non-solvent.

Of the two common processes for polyethylene production, the high-pressure, oxygen-catalysed process imposes more severe heat-transfer conditions than the lower pressure, solution polymerization. Ethylene is compressed to 1000 to 3000 atmospheres, a catalyst is injected and the reaction mixture is heated to 80 to 300°C. Low conversions make necessary a recycle of part of the unconverted ethylene which is separated from the molten polymer. It is possible to remove only a small portion of the heat of reaction through the walls of the reactor to a circulating coolant; most of it is removed by the difference in enthalpy between the feed gas and the polymer and gaseous products.

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EXPERIENCES IN FLUIDIZATION

A further three papers presented at the Symposium on Fluidization held at Adelaide, February 15 to 17, 1961.*

One paper covered work on low-temperature carbonization of coal in spouted beds in 6-in. glass and perspex columns and in a pilot carbonization plant; another paper contained details of a shallow fluid bed waste heat boiler and its fluid medium consisting of coarse and fine particles; and the third paper presented a method for the design of multistage reactors of the type required for chlorinating ilmenite.

Spouted Bed Low-temperature Carbonization of Coal

by R. H. BUCHANAN and F. MANURUNG

AN economical process for the manufacture of cheap gas, tar and power plant fuel by the fluidized low-temperature carbonization of Newston coal was the underlying motive for the research carried out by these two authors. A feature of the process investigated was the use of the spouting bed technique, which was found to be a solution to the problem of fines carry-over encountered in the experimental fluidizer.

Spouting may be illustrated by a typical air-flow/pressure drop curve obtained with a 10-in. bed of $-8+12$ mesh coal in a 6-in.-dia. perspex column. $\triangle P$ increases with air-flow up to point A, as expected in a packed column, but drops sharply thereafter. At B the air spout breaks through the surface of the bed. On reducing the air-flow the bed remains in a spouted condition up to an air velocity corresponding to point C, which represents the minimum air-flow for spouting.

In a spouted bed an entraining gas is jetted up through a bottom cone into a column of solid particles. The jet moves upwards through the bed in a central core into which the solids flow from the downward moving annulus. Solids are pneumatically conveyed up in the cone through the bed and disengage from the gas in a fountain at the top. They then fall back to the surface of the annular dense bed and move steadily down towards the cone. Some of these particles move into the cone on the way down and the balance is picked up by the spout at the base of the cone.

The work described in this paper consists of two main parts. The first deals with experiments carried out upon glass and perspex columns to obtain design data for the pilot carbonizing unit. The particular points investigated were the effect of particle sizes upon spouting and the minimum spouting velocity, the optimum inlet configuration, and the attrition of particles in the spouting bed.

Spouted Bed Studies in Glass and Perspex Columns

Experiments on the 6-in. column show that high percentages of -16 mesh fines are undesirable for spouting.

With regard to the effect of particle size on attrition, experiments indicated that the larger the particle size the greater the degree of attrition. Attrition was greater when coal was fed in with the spouting air than when it was fed in at the top of the column. However, spouting was more stable when coal was fed with the air. When no particles were introduced with the spouting gas, spouting could only be maintained if the amount of fines was small and provided the inlet tube was perfectly vertical.

The best spouting was obtained with the inlet design shown in Fig. 1. The tube protrudes $\frac{1}{8}$ in. above the flanged surface, and a gauze makes little difference to the spouting. Experiments with difficult beds, containing fines, showed that this was the only construction which would spout the bed satisfactorily.

Mathur and Gishler's equation for the minimum spouting

* For first part see *Brit. Chem. Eng.*, 1961, **6**, 259.

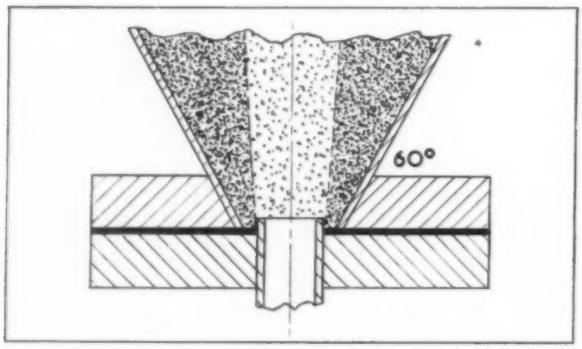

Fig. 1. Inlet design evolved for pilot carbonizer. The use of this type of inlet allowed spouted bed depths in excess of 28 in. to be obtained.

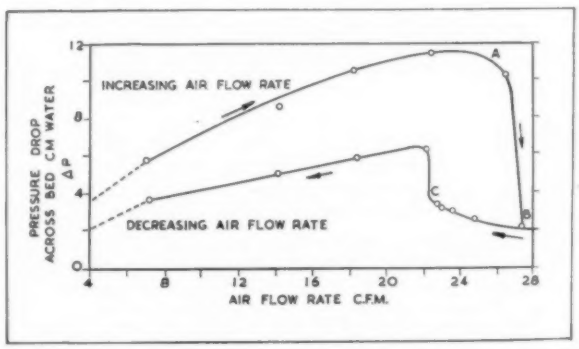

Fig. 2. Typical pressure drop is gas flow rate relationship for spouted bed.

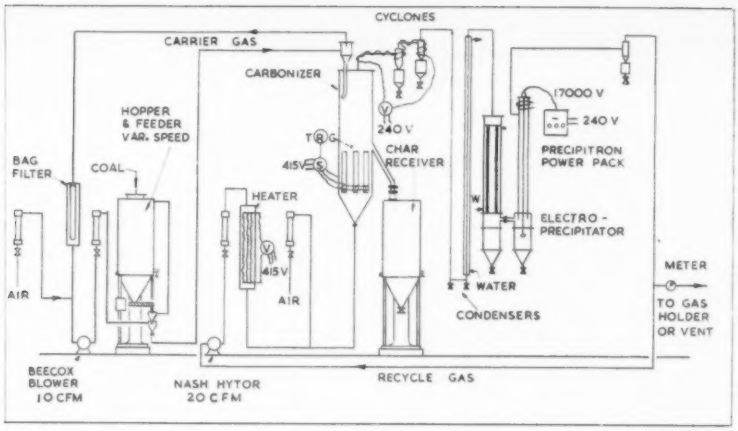

Fig. 3. Diagram of pilot plant employing spouting bed carbonization. Another feed system fed the air-coal mixture to the base of the carbonizer. The top feed system shown here was made necessary by the scarce headroom which prevented the installation of a hopper with screw feeder.

velocity derived from the wheat/air system was found to apply, to within a few per cent, to three fractions of Newston coal.

$$v_s = (D_p/D_c)\,(D_i/D_c)^{\frac{1}{3}} \sqrt{\frac{2gL(\rho_s-\rho_f)}{\rho_f}} \qquad \ldots.(1)$$

where v_s = the minimum spouting velocity, ft/sec;
D_p = the effective particle diameter (range 0.023-0.25 in.);
D_i = the inlet diameter (range $\frac{1}{16}$-2$\frac{1}{8}$ in.);
D_c = the column diameter (range 3-12 in.);
ρ_s = the solids density (range 69-464 lb/ft^3);
ρ_f = the fluid density (range 0.073-62.4 lb/ft^3); and
L = the height of the bed measured from the inlet (range 0.29-8.81 ft).

Pilot-plant Carbonization

The pilot plant shown in Fig. 3 consists of a carbonizer, feed hopper and accessories, gas system, char receiver and recovery train. The plant did not operate entirely satisfactorily and quantitative mass and heat balances could not be established.

Air alone was used as spouting gas in the early runs, in which, when the bed reached the combustion temperature, it caught fire and the temperature rise could not be controlled. Variable amounts of steam were then used in the spouting gas. The bed temperature, which, although not stated by the authors, appears to have been in the vicinity of 400°C, could be controlled satisfactorily, but the pressure drop across the bed fluctuated somewhat. Trials with 50 per cent fresh coal and 50 per cent char from the previous runs produced a more stable bed, a slight fluctuation in pressure drop, and frequent short-circuiting in the electrostatic precipitator.

One immediate aim of future work will be to devise an improved method of feeding, one which will enable coal—as distinct from char-coal mixture—to be fed satisfactorily to the reactor. The feeder in use at the time the paper was presented tended to drop the coal in slugs on to the bed rather than to distribute it uniformly with the char cycling up and down in the bed. Possible remedies are to introduce the coal with the spouting gas and accept the attrition or to feed it into the top of the fountain.

The present work, which demonstrates that it is possible to use spouting beds for low-temperature carbonization, provided agglomeration does not occur, is to be followed by further research aimed to yield more information on the effect upon spouting of a number of variables, such as particle shape and density, the "stickiness", and gas density and viscosity. In the pilot plant, work will be conducted to find out the design requirements to enable coals with caking properties to be handled, and the features of large-scale units, with multiple spouts, capable of spouting fairly large particles without high gas velocities and therefore with low power cost and little attrition.

Shallow Fluid Bed Waste Heat Boiler

by **W. J. ANDERSON**

THE main advantage of fluid bed heat transfer systems—they provide heat transfer rates of the order of 10 to 40 times greater than those encountered in conventional tubular apparatus—makes them particularly suitable for a number of difficult heat transfer applications. For example:

(1) High-temperature catalytic reactions in which the bed temperature must be held within close limits.
(2) Exothermic catalytic reactions which yield large quantities of heat, which, if recovered and used for steam generation in other parts of the plant, can realize quite substantial fuel savings.
(3) High-temperature catalytic reactions for which the reactive gas must be rapidly cooled to prevent substantial yield losses which may result from unwanted side reactions.

The author of this paper emphasizes what is sometimes overlooked; namely, that although much has been written about heat exchange in fluid beds, each application has its own design problems which resort to the available literature alone will not solve. Such factors as the height of the fluid bed, its optimum particle size distribution and the form and construction of the heat exchange unit can vary widely depending on the type of application.

This paper outlines a design of shallow, fluid bed, heat exchangers for high space velocity problems, in particular one in which about 10^6 Btu/hr were to be removed rapidly from a hot gas stream, with minimum retention time at the initially high temperatures. If possible, the heat was to be recovered by generating steam.

Investigation

The characteristic behaviour of a gas/solid fluid system is analogous to that of a gas/liquid system; viscous liquids, when aerated, show the same type of slugging as is observed with the coarse solids. It was therefore expected that the pseudo viscosity of a fluid bed could well be a fundamental measure of its performance and that beds of good fluidity would exhibit minimum viscosity characteristics.

From these concepts a model was proposed to illustrate the function of fine particles in a bed of coarse particles. The fines would tend to separate the coarse particles from one another, thus acting as a lubricant and reducing the viscosity. It was considered that the minimum fines concentration necessary would be that quantity required to coat each coarse particle with a mono layer of fines.

Using this model, and experimental work carried out on the

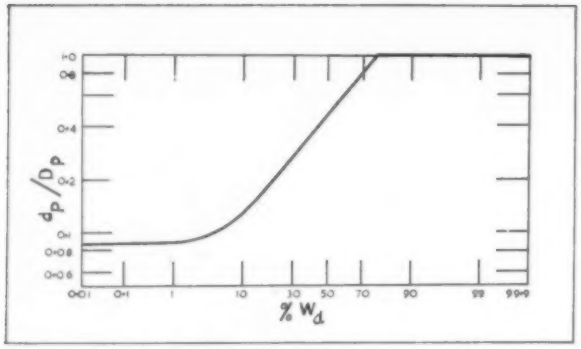

Fig. 4. Log probability plot of two-component mixtures for minimum viscosity for solving Equation (2), given average particle diameters in the fine and coarse ranges.

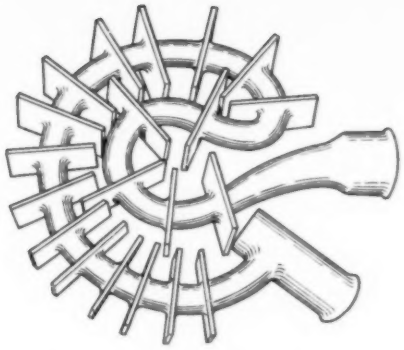

Fig. 6. Heat exchange element employing Bourdon-type tubing of approximately elliptical cross-section ($\frac{5}{32} \times \frac{11}{16} \times 0.023$ in.) with soldered-on fins, $1\frac{1}{2} \times 1 \times 0.010$ in.

viscosities of fluid beds, the following equation was derived for a two-component system:

$$W_d = \frac{1.695\left[\frac{d_p}{D_p} + \left(\frac{d_p}{D_p}\right)^{\frac{1}{2}}\right] - 0.141}{1.695\left[\frac{d_p}{D_p} + \left(\frac{d_p}{D_p}\right)^{\frac{1}{2}}\right] + 0.859} \quad \ldots.(2)$$

where d_p = particle size of fine bed;
D_p = particle size of coarse bed;
W_d = fraction of fines required.

The solution of this equation is presented graphically in Fig. 4. Thus a two-component system of good fluidity may be determined from a knowledge of the average particle diameter of the fine and coarse ranges.

Another requirement for good fluidity is that the ratio of minimum diameter of coarse particles to maximum diameter of fine particles should be $\geqq 4$ to 1.

In a fluid bed system where fines are continually lost, the fluidity of the bed is always at a maximum, since the next fraction of fines is at the required concentration. Such a bed is termed a multi-component system. Referring to Fig. 5, we see that a bed of coarse particles diameter D will retain particles of diameter greater than d. Fines having diameters smaller than d (15.4 per cent of D) will be carried out from the bed even before incipient fluidization.

In selecting the actual range of particle size of the bed, the following points must be kept in mind:

(1) Finer particles produce more effective heat exchange.
(2) Ultimate particle range depends on nature of the process.
(3) The heat transfer coefficient does not vary much with mass velocity for small particles, whereas it varies appreciably with large particles.

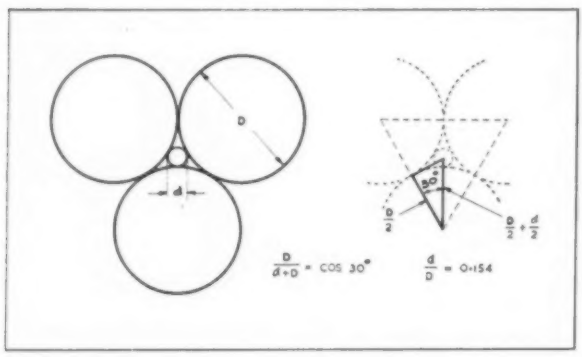

Fig. 5. Illustration showing ratio of geometric mean particle diameter to smallest particle diameter. This model of three adjacent particles each with diameters equal to the geometric mean particle size of the total bed suggests that the bed can screen particles 15.4 per cent of the geometric mean.

The shallow bed design was for a two-component system and beach sand was the selected bed material. Fines were in the 100 to 150 micron range and the range of the coarse particles was 600 to 900 micron. Therefore, $d_p = 125$ microns; $D_p = 750$ microns; and $W_d = 16$ per cent.

Electrostatic Effects

Electrostatic charges building up on the surface of the sand particles caused a lot of agglomeration. Sand was treated with calcium chloride and colloidal graphite and the latter treatment was the most satisfactory. Sand was soaked in automobile oil additive containing graphite and the oil was then evaporated using radiant heat.

Heat Exchange Unit Design

Generally, the heat exchange in fluid beds takes place either across the wall of the vessel or across an immersed internal surface. Internal surface heat exchange was chosen for this work on account of the shallow bed. Also, for internal heat transfer the maximum coefficient is reached at a lower fluidization velocity and is higher than the wall coefficients at these low velocities.

For maximum area of contact a "Bourdon gauge" type tube having elliptical shape was chosen. Fins were soldered to the tube to increase the heat exchange area. The tubing was installed horizontally in the bed and the sand added to cover the fins completely (Fig. 6). The heat transferred across the tubing wall was measured by the temperature rise of cooling water flowing through the tubes.

Bed temperatures varied widely from one coil position to another; temperatures were much lower at points where the maximum exchange surface was available. From thermocouple measurements of temperatures at different points, an average bed temperature was found for the calculation of $\triangle T$ between the bed and the tube wall.

Air at 350°F was chosen and the flow rate was varied over a four-fold range. It was found that a maximum coefficient of 87 Btu/hr sq ft/°F was obtained at 250-300 lb/hr/sq ft, which is shown to correspond to 1.7 times the minimum fluidizing velocity of the bed (165 lb/hr/sq ft).

Scaling Up

With these units it is necessary to superimpose multiple units upon one another to increase the heat transfer area. The use of the conventional heat transfer equation ($q = UA\triangle T$) proved unsatisfactory on account of the difficulty in predicting $\triangle T$. Instead, a method based on heat exchanger effectiveness versus N.T.U.[1] (number of heat transfer units) was used.

REFERENCE

[1] KAYS, W. N., and LONDON, A. L. (1958) "Compact Heat Exchangers", Chapter 2, McGraw-Hill Book Co. Inc., New York.

The Chlorination of Ilmenite

by I. B. KETTERIDGE

THE purpose of this work was to provide a method for the design of multistage fluid bed reactors required for a process of manufacturing rutile from ilmenite.

The two reactions involved are:

$$2FeO\ TiO_2 + \tfrac{1}{2}\underset{(air)}{O_2} \xrightarrow[temp]{high} Fe_2O_3 . TiO_2 + TiO_2$$

and

$$Fe_2O_3 . TiO_2 + TiO_2 + 3Cl_2 \longrightarrow 2FeCl_3 + 3/2\ O_2 + 2\ TiO_2$$

Small-scale batch tests showed that ilmenite, which in the work described was mainly of particles in the size range 72-150 mesh, required 1 hr for oxidation and 3 hr for chlorination.

Both reactions must be more than 99.5 per cent complete and a method of predicting the degree of reaction completion for reactors of the type shown in Fig. 7 was required.

The method developed is applicable to any system where well-mixed solid particles react at measurable rates with or without a fluid reagent as well as to systems such as continuous leaching of ground ores in series of stored tanks.

The method has three stages. First, frequency functions for retention times for well-mixed reactors are derived. Second, reaction rates are discussed; third, expressions for reactor efficiency developed. In general, the extent of individual particle reaction depends on its retention time in each stage of reactor.

Distribution of retention times depends on degree of mixing in each stage; tests with ilmenite in perspex models indicated that mixing was practically instantaneous and was complete.

1. Reactors with Equal Size Stages

I—Reactor Effluent

V = capacity;

v = constant flow rate;

$f(t)$ = fraction of reactor effluent retained in reactor for longer than time t, i.e., frequency distribution function of effluent.

$f(t)$ is also frequency distribution function of reactor contents.

For one-stage reactor:

$$f_1(t) = e^{-\frac{vt}{V}}$$

and frequency function:

$$\varphi_1(t) = \frac{v}{V} e^{-\frac{vt}{V}}$$

For two-stage reactor:

$$f_2(t) = \frac{vt}{V} e^{-\frac{vt}{V}} + f_1(t)$$

and

$$\varphi_2(t) = \left(\frac{v}{V}\right)^2 t\, e^{-\frac{vt}{V}}$$

General relations for a reactor of n stages:

$$f_n(t) = e^{-\frac{vt}{V}} \sum_{i=0}^{n=1} \frac{1}{i!} \left(\frac{vt}{V}\right)^i$$

and

$$\varphi_n(t) = \frac{1}{(n-1)!} \left(\frac{v}{V}\right)^n t^{n-1} e^{-\frac{vt}{V}}$$

For residence time calculations, the distribution function $f_n(t)$ can be evaluated by substituting numerical values for n, v, V and t in the expression for $f_n(t)$. However, it is apparently more convenient to make use of published tables for related functions, of which there are several: (a) incomplete Gamma function; (b) poisson distribution; (c) chi square distribution.

II—Reactor Contents

The corresponding expressions for reactor contents, for a multiple-stage reactor, can be represented by $f_{nc}(t)$ and $\varphi_{nc}(t)$

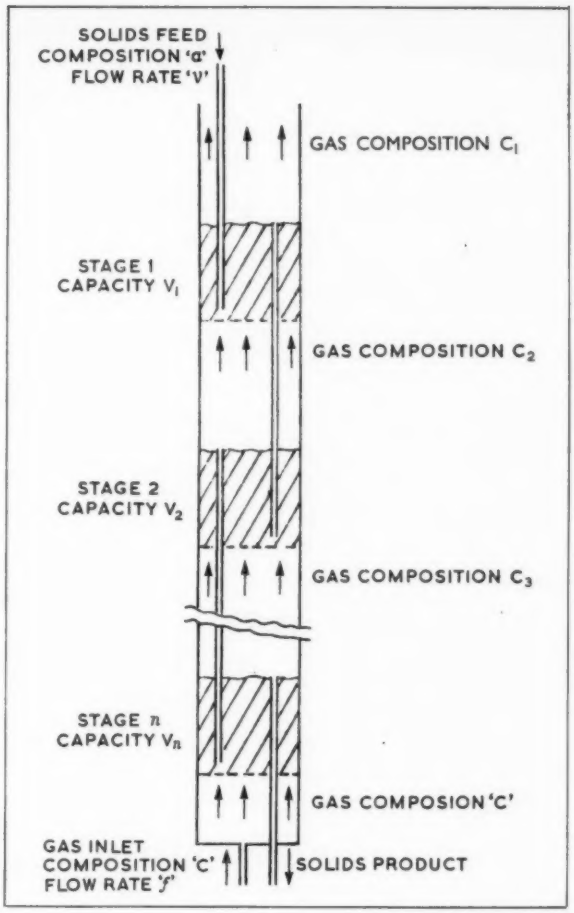

Fig. 7. Diagram of multistage fluid bed reactor. The design of reactors of this kind was studied by KETTERIDGE.

where

$$f_{nc}(t) = e^{-\frac{vt}{V}} \left(1 + \frac{n-1}{n}\left(\frac{vt}{V}\right) + \frac{n-2}{n}\frac{1}{2!}\left(\frac{vt}{V}\right)^2 + \ldots + \right.$$
$$\left. + \frac{2}{n}\left(\frac{1}{(n-2)!}\right)\left(\frac{vt}{V}\right)^{n-2} + \frac{1}{n}\left(\frac{1}{(n-1)!}\right)\left(\frac{vt}{V}\right)^{n-1}\right)$$

and

$$\varphi_{nc}(t) = \frac{1}{n}\frac{v}{V} e^{-\frac{vt}{V}} \left(1 + \frac{vt}{V} + \frac{1}{2!}\left(\frac{vt}{V}\right)^2 + \right.$$
$$\left. + \frac{1}{3!}\left(\frac{vt}{V}\right)^3 + \ldots + \frac{1}{(n-1)!} \cdot \left(\frac{vt}{V}\right)^{n-1}\right)$$

Fortunately, numerical values for frequency and distribution functions for reactor content are not often required; both these functions can be obtained from values for reactor effluent.

2. Reactors with Unequal Size Stages

I—Reactor Effluent and II—Reactor Contents

The authors derive expressions for a multiple-stage reactor where the capacities of the stages are V_1, V_2, V_3 and V_4. Relationships are soon to be established between expressions for retention times for reactor systems with different size stages and functions for which published tables are available.

3. Reaction Rates

The rate at which such reactions proceed generally depends on a number of factors, i.e., heat or mass transfer, order of

Continued on page 407

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THE GRAPHICAL CALCULATION OF CARBON AND LOW ALLOY STEEL FUSION-WELDED PRESSURE VESSEL SCANTLINGS

by J. L. RYDER

Section 2—6 per cent Knuckle Radius End Pressings (Concave to Pressure)

THIS is the second of the series of four graphs for determining pressure vessel scantlings to B.S. 1500:58. The first graph, which appeared in a previous issue,* dealt with shells; the remaining graphs are for determining the pressure-diameter-thickness relationship of other dished- and flanged-end profiles.

The graph below is drawn especially for dished and flanged ends where the inside diameter, D_i, is equal to the inside spherical radius, R_i, and the inside knuckle radius, r_i, is 6 per cent of the inside diameter. It enables the thickness (maximum $1\frac{1}{2}$ in.) of end pressings up to 120 in. diameter and 500 psi design pressure to be readily obtained. Conversely, it will show the permissible design pressure of an existing dished and flanged end of appropriate profile, provided diameter and thickness are known.

The curves are drawn to the equation

$$t = \frac{p \,.\, D_0 \,.\, K_1 \,.\, K_2}{2 \,.\, f \,.\, J \,.\, K_3}$$

taken from B.S. 1500:58, Section Three F, Equation 15.

Four necessary conditions are imposed on the curves, and these are:

(1) No allowance is made for corrosion or for thinning which takes place during forming. In effect, this means that to whatever thickness is determined from the graph should be added an allowance equivalent to the

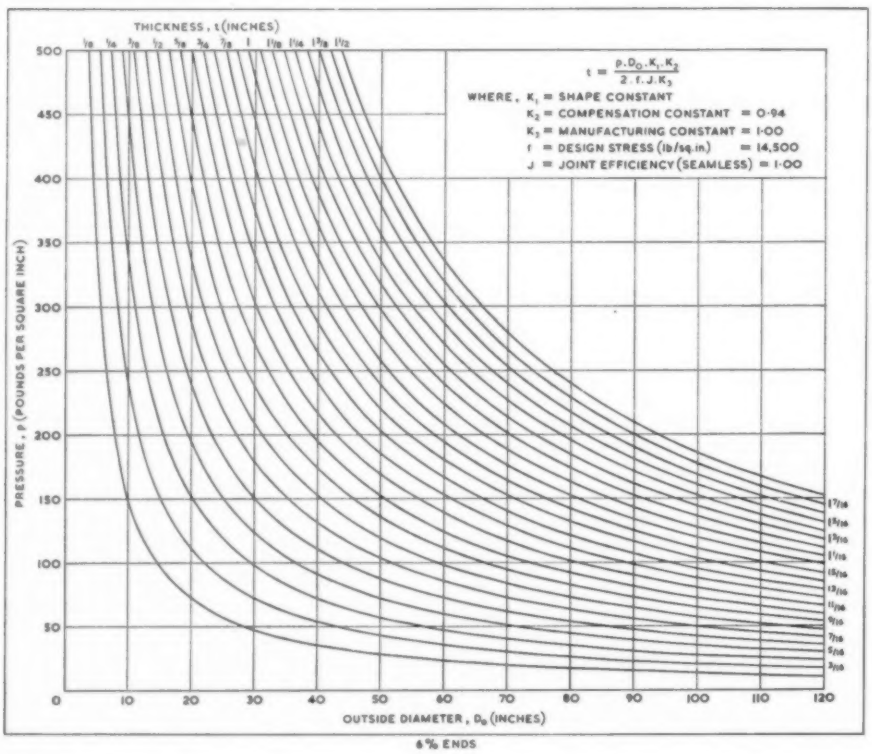

THICKNESS, t (INCHES)
1/8 3/16 1/4 5/16 3/8 7/16 1/2 9/16 5/8 11/16 3/4 13/16 7/8 15/16 1 1 1/16 1 1/8 1 3/16 1 1/4 1 5/16 1 3/8 1 7/16 1 1/2
PRESSURE, p (POUNDS PER SQUARE INCH)
500 450 400 350 300 250 200 150 100 50 0
0 10 20 30 40 50 60 70 80 90 100 110 120
OUTSIDE DIAMETER, D_0 (INCHES)
$t = \frac{p.D_0.K_1.K_2}{2.f.J.K_3}$
WHERE, K_1 = SHAPE CONSTANT
K_2 = COMPENSATION CONSTANT = 0·94
K_3 = MANUFACTURING CONSTANT = 1·00
f = DESIGN STRESS (lb/sq.in.) = 14,500
J = JOINT EFFICIENCY (SEAMLESS) = 1·00
6% ENDS

Chart for estimating the wall thickness for the dished and flanged ends of cylindrical pressure vessels. The ends employ the standard 6 per cent knuckle radius.

corrosion expected during the life of the vessel; also an additional amount to compensate for any thinning which may take place while the plate is being formed to dished and flanged profile.

(2) The material design stress is 14,500 psi; i.e., the design metal temperature does not exceed 650°F for normal boiler flanging or welding quality mild-steel plate.

(3) The end is made from a single plate without a weld seam ($J = 1.00$).

(4) Any openings in the end will be fully reinforced ($K_2 = 0.94$) and the method of manufacture shall be forming by spinning or pressing in full or ring dies ($K_3 = 1.00$).

Example

Determine the thickness of a dished and flanged end suitable for a design pressure of 150 psi and an inside diameter, new and uncorroded, of 4 ft. The corrosion allowance assumed is $\frac{1}{8}$ in. and the pressing allowance is $\frac{1}{16}$ in.

The first check shows an approximate thickness of $\frac{5}{8}$ in. plus allowances. Therefore, outside diameter

$= 48 + 2\,(\frac{5}{8}\text{ in.} + \frac{1}{8}\text{ in.} + \frac{1}{16}\text{ in.})$
$= 49.625$ in.

On entering this outside diameter and the pressure in the graph, the tentative thickness of $\frac{5}{8}$ in. is confirmed as satisfactory.

The dished and flanged end profile would be:

48 in. inside diameter;
48 in. inside spherical radius;
$2\frac{7}{8}$ in. inside knuckle radius (min);
$\frac{13}{16}$ in. thick plate.

Should a design occur where either the stress is different from that stated (i.e., 14,500 psi) or it is necessary to have a welded seam in the end pressing, due allowance can be made by applying an appropriate factor in a similar manner to that given in the article accompanying the graph for shells.*

The above information, extracted from B.S. 1500:1958, *Fusion Welded Pressure Vessels for Use in the Chemical, Petroleum and Allied Industries*, is reproduced by permission of the British Standards Institution.

* British Chemical Engineering, 1961, **6**, 3, 192.

Experiences in Fluidization

Continued from page 405

reaction, etc., and the resulting overall rate process is usually too complicated to be accurately represented by a manageable expression.

However, if particles are considered to be of the same size and composition and if temperature, pressure and other relevant parameters are constant, the rate of reaction may remain constant throughout the course of reaction or it may decrease with the change or composition of the solid particles.

In general, the rate varies as a function of the composition of the solid reactant and the function can frequently be approximated by:

$$\frac{dx}{dt} = k(a-x)^p$$

where a = initial concentration;
x = change in concentration during time t;
k = "rate" constant;
p = "order" constant.

Reaction rates have been limited to three values of p.

Type 1: $p = 1$, first order.
„ 2: $p = o$, zero order.
„ 3: $o < p < 1$, complex order.

Fig. 8 shows the course which most reactions follow for the above range of values of p.

Integrating the rate equation for the three cases gives the fraction of reactant remaining in the solid at a given time:

Type 1. $p = 1$

$$\frac{a-x}{a} = e^{-kt}, \quad o \leqslant t \leqslant \infty$$

Type 2. $p = o$

$$\frac{a-x}{a} = \frac{a-kt}{a}, \quad o \leqslant t \leqslant \frac{a}{k}$$

Type 3. $o < p < 1$

$$\frac{a-x}{a} = \left(1 - \frac{kqt}{a^q}\right)^{\frac{1}{q}}, \quad o \leqslant t \leqslant \frac{a^q}{kq}$$

where $q = 1 - p$

4. Reactor Efficiency

Values of efficiency are obtained by combining the expressions representing reaction rates for solid particles with frequency functions for particle retention times.

The general expression for heterogeneous reactions of type 1

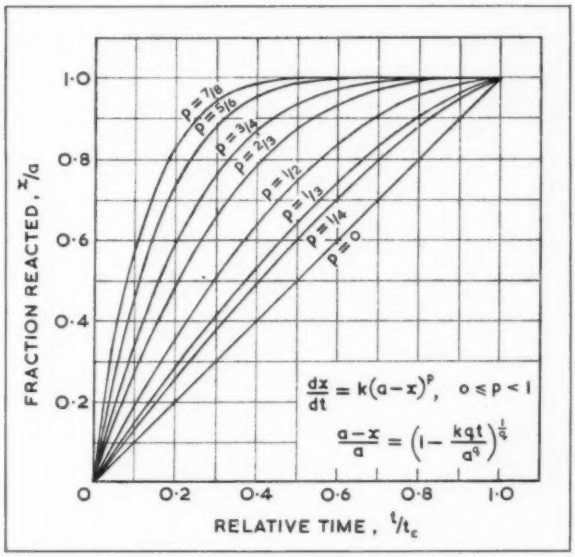


Fig. 8. A family of curves representing fraction reacted vs relative time for different orders of reaction.

is the same as for homogeneous reactions of type 1, because the extent of reaction and the probability of particle retention in any one stage are both exponential functions of time. This is not so for other types of reaction, and different expressions result.

Expressions are also derived for particles of different size but uniform composition.

The main problem which remains before the method can be applied is to correlate reaction rate constants k's and p's for both reactions, in terms of such experimental variables as temperature, gas flow rate, and gas composition; when this has been completed, it should be possible, with the aid of a digital computer, to design the required multistage reactors.

Fig. 1.

THE large number of complete plants and groupings of equipment carrying out a complete processing operation, shown by model or flow diagram, was one of the outstanding features of this exhibition, and in this domain Humphreys & Glasgow were especially prominent with their model of the ethylene plant shown above, the full-scale version of which is destined for the Leuna works. It is also a fact—and this represents something of a challenge to British manufacturers—that the planned-economy countries also had a great deal to show in the way of complete plants; and if the designs by advanced Western standards may not have been invariably novel, there was plenty of evidence of sound construction and rational layout. There is no doubt that this type of exhibit was most attractive to the visitors from countries without a developed chemical industry.

Complete Plants

Among plants for heavy chemicals may be mentioned the complete soda ash plants based upon the Solvay process offered by VEB Inex Berlin, the equipment being supplied by VEB Maschinen- Und-Apparatebau Stassfurt and VEB Maschinenfabrik Halle. Standard sizes are 150, 300 and 600 tpd of main product, but larger plants to special order can be supplied. The performance is in line with what one would expect from a well-designed plant; ammonia losses are 4 kg; and other requirements are (steam exhaust at 1.5 atm) 2t, brown coal 500 kg, power 70 kW (without auxiliary power generation), all per ton of product. The contracting organization can provide the entire soda factory, including limestone quarrying equipment, power station, workshops, administrative buildings, medical centre and so on.

Complete electrolytical plants for the manufacture of sodium hydroxide, hydrogen and chlorine from brine are one of the main fields of activity of VEB Maschinenfabrik Halle, the limit to the sizes being set by the number of cell units, an example of which was shown at this company's stand at the Fair (Fig. 7). These are mercury type with mercury pump mounted at one end of the grouping, the entire unit being designed for easy dismantling for maintenance purposes. The cell groupings are available in two sizes, one for a current load of 2500 or 5000 amps. In either case the specific loading is 3970 amps/m^2. The cathode area is 6.3 and 12.7 m^2 respectively. The output per cell is 790 and 1580 kg per day respectively of 60 per cent NaOH. The latter is produced as a 45-50 per cent solution.

In a different field oil extraction plants were offered by VEB Schwermaschinenbau Karl Liebknecht, Magdeburg. Here again three sizes of plant have been standardized, although larger units can be produced to special order. They employ benzene or hexane as extracting agent and the three sizes will treat 75, 125 or 200 tons of oil seeds per day respectively.

Solvent losses per ton of soya beans, peanuts or rape-seeds are stated to be about 7 kg, steam consumption 400-450 kg and cooling water consumption 20-24 m^3, given water at 15°C.

The same company makes also complete sugar factories, an example of a grouping it offers in this field being a juice cleaning plant designed according to the system Teschner-Ahlers. This is supplied in standard sizes capable of handling 1000, 1500, 2000 and 2500 tons of sugar beet preparation per day. The individual pieces of equipment required in such plants, such as the vacuum cellular filters, saturation vessels and thickeners which are totally enclosed vessels are manufactured at Magdeburg.

Plants for hardboard manufacture were offered by a number of exhibitors; for example, the Polish firm ZEMAK, who showed a model of a complete plant with an output of 20,000 tpa hardboard and 12,000 tpa of porous board.

Smaller Plants

Plants for making formaldehyde by catalytic decomposition of methanol upon a silver gauze catalyst are manufactured by VEB Apparate-u. Rohrleitungsbau Reinsdorf. These units were arranged in three sections—the manufacture of raw formaldehyde, the purification of formaldehyde and the recovery of methanol. An almost theoretical yield is claimed for these plants; from 100 kg of methanol is produced 190 kg of formaldehyde as solution containing 36 per cent by weight of formaldehyde and 9 per cent methanol.

Another example is VEB Chemische Maschinenbauwerke, Rudisleben/Thuringen, who undertake the manufacture of coke-oven treatment plants 5500-5800 m^3/h of N_2-H_2 mixture in the proportion 1:3 from 7500 m^3/h of coke oven gas. Methane and ethylene are also separated and may be used for other purposes, such as heating. These plants are sold as packaged units consisting of several sections such as the preliminary cooling of c.o.g., ammonia refrigeration unit for the c.o.g., high-pressure N_2 unit, and gas separation unit. Included also is a central control panel with instruments for metering the quantity of N_2-H_2 mixture. A complete set of analytical instruments is also provided. Materials of construction are copper and bronze with the parts in contact with c.o.g., etc., tinned to prevent formation of copper acetylide. The plants weigh 63 tons and can be accommodated in a building 15 × 10 × 10 m.

Heavy-duty Mixers

Of individual pieces of equipment, mixers of many kinds were in evidence and a number of novelties were to be seen. Thus for the mixing of plastic powders with stabilizers, extenders and lubricants and incorporating liquid or powdered dyestuffs, Gebr. Lödige, Maschinenbau GmbH. Paderborn, announced a new mixing layout. This consisted of two units arranged one above the other, the upper being the main mixing vessel and the other a cooling mixer. The mixing action in the upper section is provided by a combination of high-speed shearing elements and slower moving centrifugal plough-share-shaped blades which prevent build-up on the cylindrical walls of the vessel, which is jacketed for 5 atm steam. The heated mix, when judged to be complete, is transferred into the water-jacketed mixer with plough-share-type agitator elements. During the cooling a fresh charge is introduced into the main mixing compartment. The

whole is arranged with programme control with automatic feed and discharge from the upper vessel to the one beneath and to and from the unit as a whole. Four models are available with capacities based upon 60 per cent of the total volume of the upper mixer alone, of 50-570 kg, and times of mixing vary from 10-40 minutes, according to the type of feed.

In the case of the larger machine three motors are provided for driving the agitator elements, each of 5 hp, and for the cooler mixer a drive supplying 27 hp is provided. The capacity of the larger mixer is 1220 litres and that of the included cooling mixer is 1400 litres.

Other Special Mixers

A number of other new designs of mixers suitable for heavy duty were to be seen and, indeed, the presence of such machinery was another feature of the Fair.

An unusual design with a very wide field of use was shown by VEB Maschinenbau-Kombinat Freital. This is provided with a unique agitative system consisting of a ported cone and a system of scraper blades operating within and without the surface of the cone (Fig. 4). A particular advantage of these machines, one of which was shown mixing powders, is the rapidity with which a uniform mix is obtained and in this respect it is claimed to be superior to other heavy-duty mixers. Its applications range from those for which one would customarily use a kneader on one hand, and emulsifier on the other.

The sizes available range from 1-2400 litres net capacity and motors provided range from 0.63-39 kW, speeds, in most cases, being 1450 rpm.

The same company makes a combined unit consisting of two mixers one above the other. This packaged unit includes the instrument panel and switches for controlling the operations of the mixing process; the lower mixer functions as a cooler. At present this machine is made in 150 litres capacity only.

A machine shown by Draiswerke GmbH, Mannheim-Waldhof, is of quite different construction (Fig. 10), and is also suitable for heavy-duty mixing such as the mixing and kneading of viscous and pasty substances. It is a very refined version of the change pan mixer and its two agitators operate planetwise. A number of blade arrangements are available, according to the type of duty. Thus blades may have an opposed staggered spiral arrangement or may be vertical rod type.

A feature of the machine is the hood, which makes the mixing operation a totally enclosed one, which is raised or lowered hydraulically. Unlike some other change pan mixers, this one can be provided jacketed for heating or cooling purposes. The machine is available in two sizes, the hp's being respectively 7½ and 10, and capacities are 165 and 300 litres respectively. Operation sequence is controlled automatically and is actuated by push button.

A high-speed mixer for heavy duties was shown by Vaterland-Werk Neuenrade i.W. This is a tiltable mixer, thus giving easy discharge, a major feature being its ability to generate heat and for this reason it can be used as a drier. It has a water jacket for cooling purposes, and when cooling is needed the agitating system is reversed and its speed reduced. A typical performance is the raising of 40 kg of PVC compound at room temperature within 10 minutes to approximately 120°C, whereupon plasticizer can be added. Cooling from 120 to 60°C is carried out within 5 minutes. Effective capacity of the machine is 100 litres and hp's range from 7-30. Speed of impellers varies from 450-1450 rpm, the slower speed being used for the cooling operation.

Another machine to catch the eye was a glass-lined, tiltable, open agitative vessel displayed by VEB Emailleguss Radebeul. Capacity is 250 litres and a variety of mixing elements and baffles are available such as anchors, turbo or paddle types. A feature is the provision of an infinitely variable speed rotation between 250 and 330 rpm. A useful attachment is a tachometer fitted at the top end of the agitator shaft.

Pumps in Plenty

As is customary in exhibitions showing chemical plant in any quantity, pumps of many kinds were to be seen, from straightforward centrifugals for process purposes to special designs of rotary and reciprocating pumps. Multi-stage centrifugals intended primarily for drainage from mines were to be seen on the Russian stand, although they were stated to be useful for non-corrosive liquids. Maximum capacities are 70 m³/h with heads per impeller of 25-35 m of water, and a maximum number of 10 stages can be incorporated in a single pump. Shaft sealing is by deep stuffing box with lantern ring.

Another Russian pump, a free plunger variable-displacement machine designed specifically for chemicals, was displayed by Machinoexport. This seemed a straightforward, robust design with displacement adjustable while the pump is in motion. Delivery is in the range 2-4 m³/h. at a pressure of 100 atm.

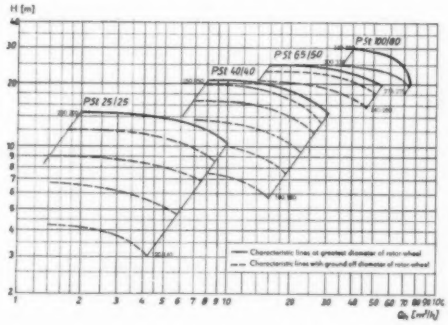


Fig. 2. Characteristics, based on water at 20°C, of porcelain centrifugal chemical pumps made by Keramische Werke, Hermsdorf.

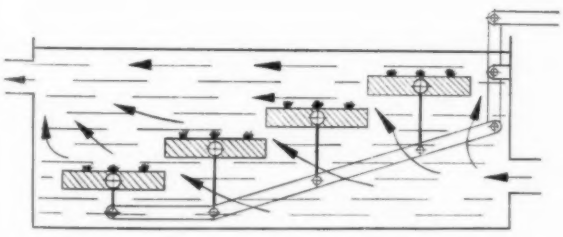


Fig. 3. A separator with magnetic filter plates with adjustable throughway shown by H. Sommermeyer Gera. Units using this principle can be totally enclosed.

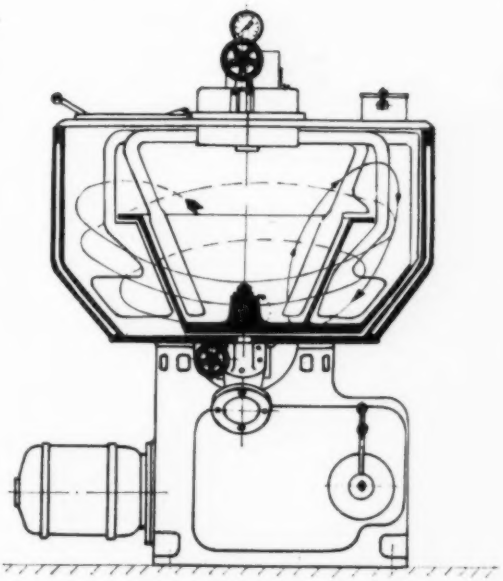


Fig. 4. Suitable for a number of arduous mixing tasks —a design of high-speed mixer shown by VEB Maschinenbau-Kombinat Freital.

A 20-kW motor is fitted and crankshaft speed on the pump shaft is 205 rpm.

A new pump shown by Lederle, designed specifically as a process pump, is available in one to three stages, the pumps being of the monobloc design. Normal rpm are 2,800 and for volatile liquids speeds up to 4000 are used. Capacities range from 10 to 800 l/m, with delivery heights of 2-84 m. Numerous materials of construction are employed, such as stainless steels, Hastelloy B or C, and for acids service a single-stage rubber-lined construction is also available.

Pumps employing the Mono principle were shown by Seeberger K.G. One model for difficult pumping duties is mounted in a short, narrow cylinder attached to the conical base of a small tank, the exit from the tank being common with the pump discharge. The drive, which includes variable-speed gear mounted at the top of the vessel, is connected to the pump rotor by a vertical shaft. The range gives pressures at maximum revs. of 4.5 and 9 atm respectively, the deliveries varying from approximately 1780 to 11,500 l/h respectively. The capacity of the receiver above the pump rotor varies according to size of pump from 150-500 l. This arrangement has been devised for materials with poor flow properties which normally would be very difficult to feed into the inlet end of any type of pump.

Pumps of more conventional layout made by this company were shown with stators made of PTFE, natural rubber, viton, butyl rubber, nylon, polyethylene, perbunan, hypalon, neoprene, thiokol and silicones.

Another pump shown by the same company was a twin rotor model with the rotors arranged in parallel. Maximum throughput is in excess of 60,000 l/h for pressures of 4.5 atm and in excess of 50,000 for pressures of 9 atm; it has a better suction performance than the single rotor type and the lift with water is 7.6 m.

Valves with Actuators

Like pumps, valves were present in abundance, the types to be seen ranging from manually-operated porcelain plug cocks to valves actuated by pneumatic, mechanical or hydraulic means. Valves of this kind were shown by two British firms in particular, Newman Hender and Limitorque Controls. The valves shown by the former company were fitted with Rotork electrically-operated actuators and included a 24-in. lubricated parallel plug valve and a 16-in. heavy sealing gate valve. The exhibitor emphasized the suitability of this equipment for fully automated process plants. Among the equally impressive Limitorque display was a range of automatic valve control units operable by virtually any power source. An electrically-operated controller was shown attached to a few large valves including a 12-in. gate valve (Fig. 9).

Valve actuators were also shown by West and East German firms, in particular VEB Messgeräte- und Armaturenwerk Karl Marx, Magdeburg-Buckau, and VEB Armaturenwerk Reichenbach. Forged alloy steel valves fitted with electrical actuators or hydraulic actuators were shown by the latter firm attached to 4-in. right-angle valves. These were specially designed for synthesis plants and are capable of withstanding pressures of up to 700 kg/cm^2. Sizes range up to 120 mm.

Mechanical Separation Apparatus

The various types of mechanical separation equipment are invariably well supported at this exhibition, and such household names as Krauss Maffei, Alfa-Laval, Westfalia and others usually occupy a prominent position.

Of the pressure filters shown, one of the most interesting came from VEB Maschinen- Und Apparatebau, Stassfurt, which, from the diagrams presented, seemed a very serviceable unit and bore a resemblance to the streamline filter. It is a vertical unit with a number of cartridge-type filter elements which consist of cylindrical rings made of either ceramics, porous plastic, carbon, sintered metal or glass, threaded upon perforated steel tubes. The feed is at the bottom and discharge at the top. The upper end is also provided with a channel for the introduction of air or flushing liquid. Two types of base are fitted, depending upon whether the cake is to be dried or merely flushed from the filter medium. In case of the former the cake is blown off by compressed air and discharged by means of a hinged cover which is lowered by handwheel-operated gearing. If the cake is to be flushed by liquid, a screw discharge is fitted instead.

Air-swept Sieve

One of the novelties in the separation field was an Alpine A.G., Augsburg, unit for sieving powdered solids. The material to be sieved is fed into an inclined rotating cylindrical sieve and fine material is removed by an air stream drawn in through a narrow port extending the entire length of the cylindrical sieve. The sieving action is helped by the entry of air into the sieve compartment which entrains the fine particles and removes them through the sieve, the coarse material being discharged at the lower end of the sieve. The air velocities on either side of the sieve mesh are adjusted so that the material does not blind the mesh openings. Nevertheless, a sieve clearance device is provided; the fine material is collected directly from the sieve housing and the air passed through a filter system. In some cases both air and product can be treated together in a filter system.

The enclosed sieve is completely visible through a long window on its housing, and is driven at 50 rpm by a fractional hp motor. The total pressure drop is 420 mm wg, and the air volume is 600 m^3/h; time of sieving is 1-2 min and mesh openings of 33, 44, 60 microns and greater are available. For sieving materials down to 20 microns a precision micro sieve can be provided.

Instruments

Instruments from the G.D.R. focused attention upon the unitized system for electrical and pneumatic instruments now widely used in the planned-economy countries. According to this system, instruments and controller input and output values are standardized, as well as the dimensions of components and connections which make up a complete control system. Examples of equipment conforming to these standards were shown by VEB Messgerätewerk Quedlinburg and VEB Geräte- und Regler-Werke Teltow. The former showed three term controllers of pneumatic or combined electronic-pneumatic types and, in addition, a pneumatic computer capable of manipulating two variables in a number of combinations.

The Teltow company deployed a wide range of equipment, including systems for controlling conductivity (Fig. 8), level, ratio, volume and humidity. In addition, the same company showed a control piston for the electrically-operated remote control of hydraulic systems, and a converter unit for transforming a measured electrical value into a proportional pneumatic one.

Also much in evidence were programme controllers, and VEB Intron Leipzig demonstrated a magnetic tape control mechanism for regulating high-speed machinery, as well as a transmitter for controlling the magnetic control vanes of electro-hydraulic systems.

Russian exhibits in the field were scarcely directed towards the process industries, but an exhibit by Mashpriborintorg consisted of a computer used for the control of temperature at 200 different points.

Other novelties included a magnetic flowmeter by J. C. Eckardt which can be supplied with an electro-pneumatic control system and in bores up to 1 m and lined with rubber, PTFE and glass, etc.; and apparently the only one of its type to be shown, a photometric instrument manufactured by Georg C. K. Withof GmbH, Kassel, which can be used for either gases or liquids provided they have a specific absorption for visible or ultra-violet light. Ranges from 0-0.1 to 0-100 per cent for instruments operating with visible light and from 0.01 to 0-100 per cent by volume when ultra-violet light is used. Accuracy is 3% of the measurement range.

Materials of Construction

Equipment in non-ferrous and non-metallic materials was plentiful, although nothing startlingly new appeared. Pumps, valves, pipework, packed and bubble plate columns and agitated vessels in stoneware and porcelain were on view. The Hermsdorf works, for example, showed a packed column for vacuum operation of diameter approaching 1 m. with a perforated, dished, vapour distributor. Glassware was represented by Jenaer Glaswerke Schott and Gen. with a number of exhibits similar to those of last year, such as a 500-mm. bore bubble or packed column supplied in heights of 20 m. or greater for distillation. For distillation these columns operate with multiple double-coil reboiler units.

Glass tiles for corrosion-resistance or freedom from product-contamination were shown by Säurebau Byczkowski K.G.

Fig. 5. An hydraulically-operated process valve for high-pressure synthesis shown by VEB Armaturenwerk Reichenbach.

Fig. 6. A glass-lined alkylation vessel destined for Leuna shown by VEB Eisen- und Hüttenwerke Thale. In the foreground a new 630-litre glass-lined reactor from VEB Emailleguss Radebeul.

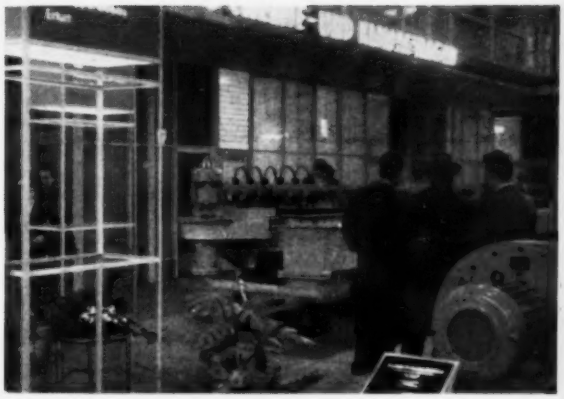


Fig. 7. A mercury cell for the manufacture of caustic soda from brine, capable of giving a product of 50 per cent strength. Shown by VEB Maschinenfabrik Halle.

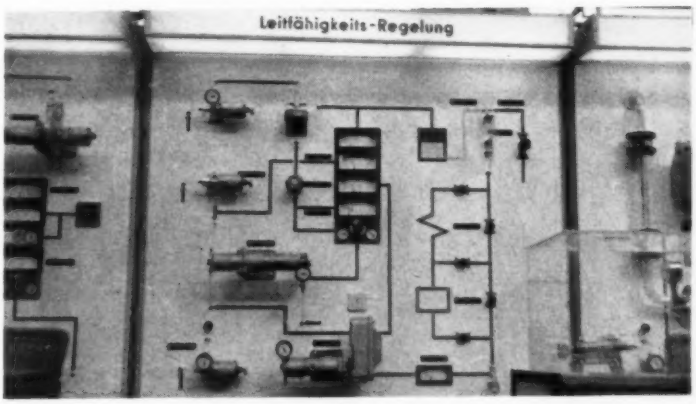


Fig. 8. Conductivity control equipment shown on the stand of VEB Geräte- und Regler-Werk Teltow.

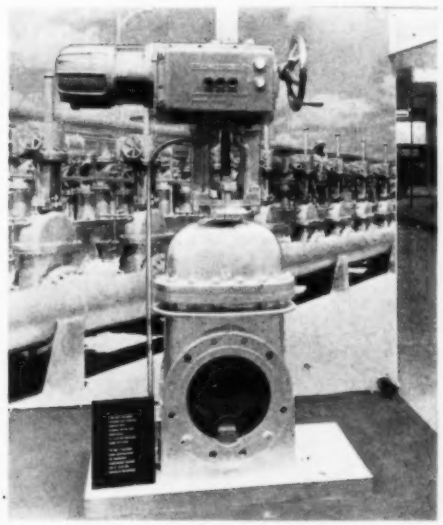


Fig. 9. A view of the Limitorque stand; an electrical control unit made by this company attached to a 12-in. Alley & McLellan gate valve.

Fig. 10. One of the heavy-duty mixing machines shown by Drais—a refined change pan mixer with planetary motion agitators.

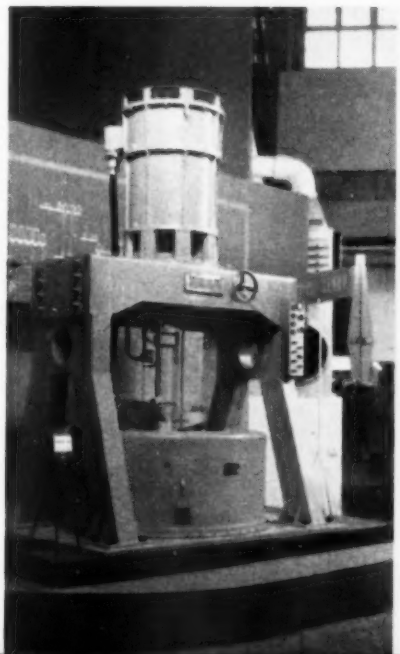


Fig. 11. A scene in the Polish pavilion —a sugar centrifuge shown by Cekop. The machine has four speeds: 50, 200, 750 and 1470 rpm.

Fig. 12. Automatic process control components shown by Honeywell Brown, Frankfurt.

Abstracts

GROUP A — FLUID DYNAMICS

The Upward Vertical Flow of Oil-water Mixtures

These experiments were made in a 1.038 in. I.D. 37-ft length of cellulose acetate tubing. Flow pattern, hold-up and pressure drop data were obtained for oil-water mixtures at superficial water velocities from 0.1 to 10.0 ft/sec.

The pressure drop due to friction and other irreversibilities is essentially independent of the viscosity of the oil, except where the oil is the continuous phase.

G. W. Govier *et al.*, *Can. J. Ch. E.*, 1961, **39** (3), 67-75.

The Reduction of Pressure Gradients in Oil Pipelines

This paper describes the effect of pumping water with a black crude oil (sp. gr. 0.9 and $\mu = 545$-1200 cP) in 1-in. and 2.5-in. pipes. Stratified flow was obtained with approximately 50 per cent water and the pressure gradient was reduced by a factor of 10. Emulsions formed with less water. The possibility of commercial application is discussed. In a long line, oil and water would have to be separated at each pumping station.

M. F. Charles, *Can. Min. Met. Bull.*, 1960, **53**, 483-7.

Control of Surges in Liquid Pipelines

A method is presented for calculating the magnitude of pressure surges in pipelines. Air pockets trapped in pipelines may lead to dangerous surges.

Anon, *Pet. Eng.*, 1960, **32** (10), D 60.

Experimental Study of the Flow Conditions and Pressure Drop of Steam-water Mixtures at High Pressures in Heated and Unheated Tubes

Part I describes the high-pressure experimental plant and its associated instrumentation. Special differential manometers and thermocouples were developed for the accurate measurement of small differences in pressure and temperature at high fluid pressures.

Part II deals with the analysis of experimental results obtained from measurements of the absorption of X-rays in their passage through the two-phase mixture at outlet from the test section.

Part III presents an analysis of the pressure-drop measurements made on the 1-in.- and 1½-in.-bore pipes in both the horizontal and vertical positions.

R. W. Haywood *et al.* A paper presented at an Ordinary Meeting of the Institution of Mechanical Engineers, in London, on March 29, 1961.

Drag Forces in a Hydraulic Model of a Fluidized Bed—Part 1

The paper described experiments carried out to measure the drag on a sphere (½ in. to ⅝ in. dia.) in a water stream (Reynolds No. 10-1000) and the effect which neighbouring spheres had on the drag. The drag on a sphere in a packed array is about 70 times that on a similar isolated sphere. Adjacent particles repel one another, but, when spheres are in line, they reduce the drag on one another.

Some of the features of fluidized beds are explained, but further knowledge of the gas flow patterns is needed before these results can be applied to bubble formation in gas fluidized beds.

P. N. Rowe and G. A. Henwood, *Trans. Inst. Ch. E.*, 1961, **39** (1), 43.
This is to replace the abstract of the same title published in May, Brit. Chem. Eng., **6**, 334.

Find Line Pressure Drop by Nomograph

A nomograph is presented which will produce the following information: (a) pressure drop in a given line; (b) line size giving a desired pressure drop; (c) line size giving economic pressure drop (for gases only); (d) pressure drop in a new line size; (e) allowable flow in a given line; (f) correct for a different friction factor.

This nomograph solves the Fanning equation expressed as:

$$\Delta p/100\,\text{ft} = \frac{f\,W^2}{74{,}000\,\rho\,d^5}$$

J. D. Lewis, *Pet. Ref.*, 1961, **40** (3), 169-70.

Liquid Viscosities at High Pressure

A graphical correlation shows the effect of pressure on viscosity. The ratio of viscosity at high pressure to the viscosity at 1 atm is dependent upon pressure, the viscosity of 1 atm and the characterization factor, Kw, of the petroleum factor.

F. J. Lockhart and J. M. Lenom, *Pet. Ref.*, 1961, **40** (3), 209-10.

GROUP B — HEAT TRANSFER

Boiling Burn-out at High Pressure in Horizontal Annuli

The authors present boiling burn-out test results for forced circulation, bulk boiling heat transfer to high purity water in a 0.192-in. horizontal annulus of 1.43 in. I.D. and 21 ft heated length. They describe the electrically heated equipment used. Test conditions were: pressure from 590 to 2010 psia, heat flux from 133,000 to 378,000 BTU/hr/sq ft, mass flow rates from 0.61×10^6 to 1.7×10^6 lb/hr/sq ft and outlet steam qualities up to 44.0 per cent by wt.

D. J. Foley *et al.* Paper presented at the Reactor Heat Transfer Conference at New York, November 1956.
TID-7529 (Pt. 2) available from: Technical Information Services Extension, P.O. Box 1001, Oak Ridge, Tennessee.

Industrial Heat-transfer Media for Temperatures up to 400°C

Heat-transfer media are described which permit a temperature of 400°C, at low pressure, to be reached. One of the most suitable media is Diphyl (Bayer trade-mark), a mixture of diphenyl and diphenyl oxide. Special heaters have had to be developed to use Diphyl.

J. Szymkowiak, *Chemie Ing. Tech.*, 1961, **33** (4), 243-5.

The Prospects for Nuclear Process Heat

Nuclear reactors will now produce low-temperature process steam economically. It is up to the process industries to extend the use of low-temperature steam in the future.

J. H. Wright and D. R. Smith, *Chem. Eng.*, 1960, **67** (24), 123-8.

GROUP C — MASS TRANSFER

Solid-liquid Equilibria in Wax Crystallization

A theoretical study of equilibria obtained during crystallization of waxes has shown that the degree of separation between normal paraffins may be predicted, if the solid is assumed to be an ideal solution. Results predicted from the theoretical equations agreed closely with mass spectrometer analysis.

Ketone solvents have a selectivity similar to *n*-hexane for recrystallizing waxes. Multi-stage counter-current recrystallization gives sharper separation of *n*-paraffins than single stage. Mixtures of similar molecular weight *n*-paraffins and waxes form solid solutions when cooled. Mixtures of *n*-paraffins of different molecular weights, or hydrocarbons of different structure, form separate crystals and a eutectic.

R. M. Butler and D. M. MacLeod, *Can. J. Ch. E.*, 1961, **39** (2), 53-63.

Diffusion in Metals

This review on diffusion in metals includes 87 refs. and covers diffusion of pure metals, alloys, surface diffusion and diffusion in liquids.

P. G. Shewmon and G. R. Love, *Ind. Eng. Chem.*, 1961, **53** (4), 325-8.

Turbulent Mass Transfer in Liquid Streams

This paper presents a study of the diffusion of a dye solution from a point source into water in turbulent pipe flow (N_{Re} 3000 to 10,000).

Total diffusivity varied considerably with radial position and reached a maximum away from the tube centre at $N_{Re} > 5000$.

The turbulent $N_{Sh} > 1.0$ for $N_{Re} < 10{,}000$ and it decreases with increasing N_{Re}. This indicates that there is not a complete analogy between mass and momentum transfer in this range.

R. C. Seagrove and R. W. Fabien, Dept. of Chemical Engineering, Iowa State University, Ames, Iowa.

Relating Theoretical Plates and Transfer Units

A one-step method is suggested for converting theoretical plates into transfer units within experimental accuracy. An example is worked out for the enriching section of a binary separation.

A. J. Surowiec, *Ind. Eng. Chem.*, 1961, **53** (4), 289-92.

Brit. Chem. Eng. Abstracts: **FOLIO No. II**

GROUP D — PHYSICAL SEPARATIONS

Batch Distillation of Binary Mixtures Provides Versatile Process Operations

A report on the design and use of batch stills for binary mixtures. The report covers column design and methods of operation—constant or variable reflux. In a column of few plates constant reflux is advantageous; with many plates variable reflux is chosen. Calculations are included for the system ethanol/water, (a) at constant reflux $R = 3.0$ in columns having 3 to 9 plates, and (b) at variable reflux in columns having 6 to 10 plates. Graphs are also presented showing the relationship of distillation rate to N.T.P. for variable reflux and a constant reflux, for the mixtures benzene/toluene, isopropanol/water, acetone/water, ethanol/water.

B. Block, *Chem. Eng.*, 1961, **68** (3), 87-98.

Liquid-liquid Equilibria*

Principles of ternary liquid-liquid equilibria are reviewed as applied especially to solvent extraction. Emphasis is placed on the profound effects of impurities or extra components, the unusual graphs which may result with certain combinations of fairly common reagents, and certain theoretical restrictions on shapes of ternary graphs, which are frequently overlooked.

These principles are illustrated with graphs of observed systems.

A. W. Francis. Paper No. 54, presented at the 44th National Meeting of the American Institute of Chemical Engineers, February 1961, at New Orleans.

Filterability of Distillate Fuels. Part I—Development of Methods and Effects of Filter Porosity

Two filtration methods were studied—a constant-flow differential-pressure method and a constant-head gravity-flow method. The constant-head method appeared more desirable; the apparatus and procedure are described.

Cellulose ester filters gave no advantage and a single layer of 10-micron paper gave satisfactory results. The average calibration time was 25.0 sec/100 ml; the determination of calibration times were repeatable to within ± 0.2 sec.

Temperature has a marked effect on both the calibration time and plugging volume. Another report will deal with this aspect.

A. J. Chiantella and J. E. Johnson, U.S. Naval Research Laboratory, Washington, D.C. NRL Report 5565.

Distillation Design with Varying Volatility

A new distillation design procedure for cases where the relative volatility varies from top to bottom trays is now extended to include multi-component systems. The equations can be solved with slide rule and desk calculator.

F. W. Winn, *Pet. Ref.*, 1961, **40** (4), 153-5.

GROUP E — PROCESSING

High-pressure, Liquid-phase Hydrogenation of Pyrocatechols

Plant tests show that high-pressure liquid-phase hydrogenation of a pyrocatechol is influenced by its boiling point and chemical composition. Most suitable for hydrogenation are phenolic-aromatic tarry intermediate products, obtained from the production of liquid fuels, which contain 50 per cent of components boiling up to 320°C. Low boiling solvents and n-alkanic naphtha residues do not suppress thermal polymerization but limit coking. By a recycle procedure, the yield of monovalent phenols is increased and concentrated phenolic fractions are obtained.

(This paper is a continuation of a study published earlier in *Chemicky Prumysl*, No. 9, p. 453 (1959).) 9 refs., 5 diagrams including 2 flow diagrams.

O. Svajgl, *Chem. Prum.*, 1961, **11** (2), 66-71.

Studies on the Selectivity of Zirconium Phosphate used as Ion Exchanger

The paper describes preliminary experiments on the effect which heating, to various temperatures, has on the selectivity of zirconium phosphate used as an ion exchange. Zirconium phosphate with cations fixed at room temperature was first heated. This selectively increased the elution resistance of some adsorbed cations. Investigations, over a wide range of temperature and heating time, have shown that the selectivity (particularly cæsium) can be altered by heating the zirconium phosphate and then fixing and eluting cations at room temperature. This selectivity remains even after the ion exchanger has been regenerated several times. High-temperature treatment markedly reduces the exchange power.

Anon. *Prodotti Nuovi d'Industria* (*Italy*), April 1960, pp. 8-9.

* Author's abstract.

Recovery of Ammonia by Staking Burnt Lime with the Liquid from a Distiller Pre-heater

A laboratory-scale investigation of ammonia recovery in soda manufacture. The reactions taking place in the lime mixer are examined, including the effect of using either a lime slurry, or raw lime obtained from the calcining of chalk. Also examined was the effect of adding NaCl upon the quantity of ammonia evolved. It is shown that burnt lime gives 20 per cent greater recovery of ammonia than lime slurry.

S. Kaminski and E. Pischinger, *Przem. Chem.*, 160, **39** (12), 759-61.

Manufacture of Wet-process Phosphoric Acid

The technology of manufacture of wet-process phosphoric acid has advanced rapidly, but there is a need for further research. The reaction of phosphate rock and sulphuric acid is carried out in an aqueous digestive system. Phosphoric acid is separated from the resulting slurry by filtration and countercurrent washing. It is important to achieve rapid filtration rates, high recovery of phosphorus as acid and the maximum concentration of acid.

Control of the size and shape of the calcium sulphate crystals is a primary factor in filtration rate and washing efficiency. Three crystalline forms are possible. The anhydrite ($CaSO_4$), semihydrate ($CaSO_4 . \frac{1}{2}H_2O$) and dihydrate ($CaSO_4 . 2H_2O$). Most processes in the U.S. produce the dihydrate, but recent developments in Europe indicate that anhydrite or semihydrate processes may have important advantages.

The factors affecting the crystal form, size and shape are reviewed. Experimental data are given to show the effects of some of the impurities in the phosphate rock and of the concentration of excess sulphuric acid in the solution. Promising areas for further research are outlined.

T. P. Hignett. Paper No. 5, presented at the 44th National Meeting of the American Institute of Chemical Engineers, February 1961, at New Orleans.

Nitric Acid Concentration by Magnesium Nitrate*

Magnesium nitrate solution is used to replace the conventional sulphuric acid in an improved process for concentrating nitric acid. Sixty per cent nitric acid produced by the ammonia oxidation process is concentrated by extractive distillation to obtain a product of 99-99.5 per cent HNO_3. The fractionating column features specially designed trays. The magnesium nitrate solution is recycled in the process and is concentrated by vacuum flash evaporation. Process advantages are high yields of pure, highly concentrated product, low investment cost, low operating cost, long on-stream time with reduced maintenance, and elimination of air pollution problems that are usually associated with nitric acid concentration.

R. J. Bechtel. Paper No. 28, presented at the 44th National Meeting of the American Institute of Chemical Engineers, February 1, 1961, at New Orleans.

Producing Hydrogen

The author compares five methods of manufacturing hydrogen: I: Steam reforming using natural gas or paraffins: $CH_4 + H_2O \rightarrow CO + 3H_2$. II: Partial oxidation of hydrocarbons to form H_2 and CO. III: Water gas reaction with coke: $C + H_2O \rightarrow H_2 + CO$. IV: Electrolytic hydrogen manufacture. V: Treatment of off-gases from coke-ovens, refineries, etc.

G. R. James, *Chem. Eng.*, 1960, **67** (25) 161-6.

Nitrogen Removal: Its Cost and Profits

A cryogenic process, employing feed gas drying, gas liquefaction, product fractionation and revaporization of both the product and waste gas streams, has been designed for removing N_2 from natural gas. Several graphs are presented to evaluate the costs and profits of this process.

D. B. Jameson and L. R. Mann, *Pet. Ref.*, 1961, **40** (4), 121-2.

Safely Operating Low-temperature Plant

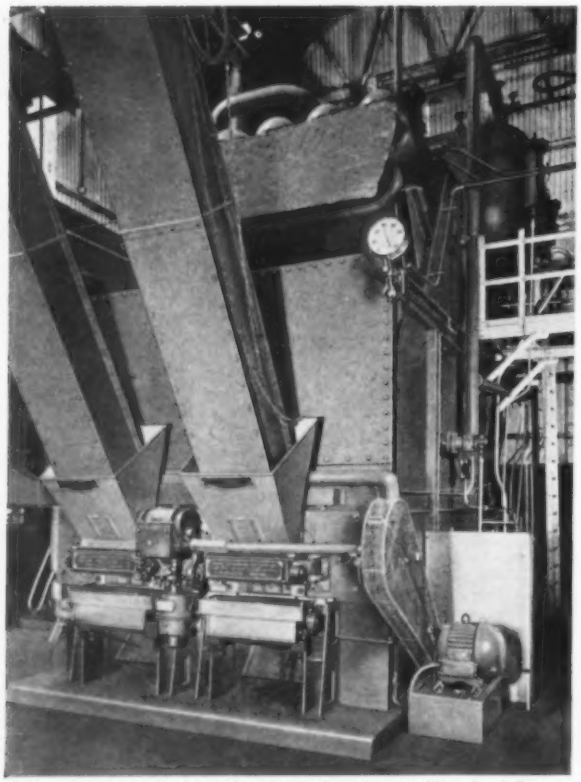
This report consists of a series of papers by, and discussions between, operating personnel responsible for safely operating low-temperature air separation and ammonia plants. The subjects covered by the papers are: (a) control of hydrocarbon impurities in air separation plants; (b) an analysis of the causes of explosions in two oxygen plant units; (c) an explosion and an incident in nitrogen-wash units; (d) a hydrogen flame ionization detector for accurate checks on contamination level; (e) a condensed survey of operating practices at a number of U.S. air and ammonia plant operators.

F. G. Kerry *et al.*, *C.E.P.*, 1961, **57** (4), 37-60.

Brit. Chem. Eng. Abstracts: **FOLIO No. 12**

BCE 9783 for further information

COAL—properly used—is the cheapest fuel for the Chemical Industry



Coking stokers feed coal continuously, quietly and invisibly into this modern boiler.

Search the industrial scene—and it is difficult to find any major industry that does not use chemicals. But even in the prospering chemical and chemical processing world, sound economics are of primary importance and wise managements will always make every possible saving and every possible improvement that will help keep prices stable while satisfying—and expanding—their home and overseas markets.

BEGIN AT THE BEGINNING—THE BOILERHOUSE

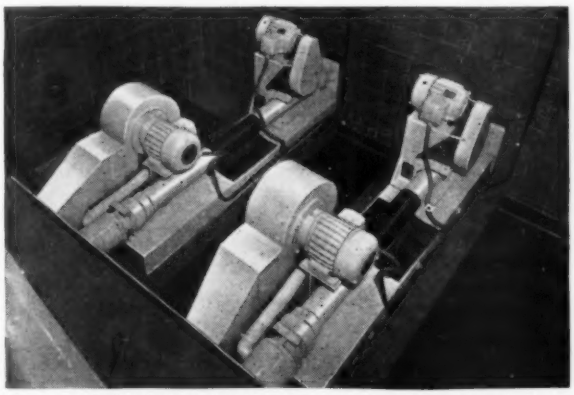
There are still some chemical plants that handicap their finances with outdated boilerhouses. To modernise other machinery while 'making-do' with inefficient boilers is to throw good money down the drain. Modern coal-burning installations can be so efficient that they will more than pay for themselves in a remarkably short time. They are, without question, the *first* essential in a chemical plant that is going to make rising profits for its management.

COAL IS THE RELIABLE FUEL

One question for any long-term planning is obvious: can supplies of coal be guaranteed? The answer is yes—without qualification. The mining industry—Britain's *largest single industry* is becoming increasingly efficient; its equipment is the best of its kind. And British coalfields contain enough coal to supply every industrial demand for centuries to come.

COAL IS CLEANER, HOTTER

Modern grading and washing methods ensure that coal is the most efficient and consistent fuel for every type of boiler—the ideal fuel for every section of the chemical and chemical processing industry. But to obtain the maximum heat at the lowest cost, coal should be mechanically stoked. A mechanical stoker automatically feeds the right amount of coal for the boiler load even when the load is varying; it burns coal at optimum efficiency all the time without the emission of smoke; and it virtually replaces the human element in boiler operation. There are many types of mechanical stokers, of which the most commonly used are the Chain Grate stoker, the Coking stoker, the Underfeed stoker and the Sprinkler stoker. Standard models are available for all sizes of boiler.



Two underfeed stokers feeding coal direct from a storage hopper into a modern vertical boiler.

OVER 80% OF BRITISH INDUSTRY USES COAL

The National Coal Board is determined that COAL shall continue to be plentiful, shall continue to keep our industrial power both dominant and progressive. You can depend upon COAL; its sources and supply are in our *own* hands, free from the fluctuations of foreign policies. Endless research by scientists and chemists working in the laboratories of the N.C.B. will ensure that coal will be increasingly profitable for its users.

DELIVERY—ON THE DOT —ON THE SPOT

The delivery of coal is today highly organised and completely reliable. Leading coal merchants have completely overhauled delivery and storage systems and are fully equipped to make sure that you get the right grade of coal when you want it and where you want it. And handling can be fully automatic. Furthermore, completely mechanised handling systems will transport coal from storage to furnace without any manual labour at all.

AND—WHEN YOU NEED IT— A NATION-WIDE FREE SERVICE

The vast resources of the National Coal Board are no further away than your telephone. At a flick of your finger, you can call upon one of the most comprehensive and efficient technical services in the world. No problem is too small or too big. You may want advice on coal quality, coal storage or coal-burning appliances or even on the siting of a new boilerhouse—whatever it is, a 'phone call or letter either to your coal supplier or to the Regional office of the National Coal Board will bring an authoritative answer to your problem.

FINANCE PLAN FOR INDUSTRY

No deposit, repayments spread over five years, low interest charges that can be set against Tax, full investment and other capital allowances, that can be claimed at once, these are the terms available to the manufacturer or businessman who wants to install modern coal-burning equipment without touching his capital resources.

Make use of the National Coal Board Industrial Finance Plan. The loans are made by Forward Trust Limited, who are members of the Midland Bank Group. A 'phone call or a letter to the Regional office of the National Coal Board will bring you full details.

SOLID FUEL—more heat at less cost—and it's British

PROGRESSIVE INDUSTRY IS GOING FORWARD ON COAL

ISSUED BY THE NATIONAL COAL BOARD

Abstracts

GROUP E — PROCESSING (contd.)

Concentration of Diamonds

Three flowsheets dealing with the subject are presented.

Flowsheet No. 1 is typical for small to medium tonnages of alluvial feed (5-30 tons/hr). It incorporates a jaw crusher, a trommel with scrubbing screen, centrifugal diamond pans, another trommel screen and mineral jigs.

Flowsheet No. 2 was developed for diamond recovery from Kimberlite ore as mined, and is suitable for up to 50 tons/hr of ore. Ore is crushed, screen and oversize material crushed again. It is then processed through a washing and scrubbing trommel, centrifugal diamond pans, vibrating screens and mineral jigs.

Flowsheet No. 3 illustrates a more complex diamond recovery method which can be used to process up to 500 tons/hr of Kimberlite ore and is also arranged to handle weathered or soft ores. It incorporates primary and secondary crushers, magnetic separator, densifier, vibrating screens, float screen and sink screen, etc.

Denver Equipment Co.'s Publication: *Deco Trefoil*, 1961, **25** (1), 13-16.

Pumping

This review describes some recent developments in the design of pumps for the chemical and process industries, with particular reference to pumps designed to resist increasingly stringent conditions.

A. Flindle, *C.P.E.*, 1961, **42** (4), 175-7.

Citric Acid

A description of the Bzura chemical process for manufacturing citric acid by fermentation of blackstrap molasses.

L. A. Agnello and R. J. Kieber, *Ind. Eng. Chem.*, 1961, **53** (4), 253-8.

Nitration of Orthomononitrotoluene

The experimental results are presented of work undertaken to provide data to show the relative importance of the major process variables, including the composition of the mixed acid, the phase volume ratio, space velocity and reaction temperature.

A miniature jacketed kettle provide absolute chemical reaction rate data. The nitration of *o*-nitrotoluene was carried out in a continuous flow system. 19 refs.

K. A. Kobe and J. T. Fortman, *Ind. Eng. Chem.*, 1961, **53** (4), 269-74.

Separation of Sulphonic and Sulphuric Acids with an Ion Exchange Resin

A process is described for the separation of two strong acids of differing molecular size, based on the screening effect of a highly cross-linked ion exchange resin. Only water is required for regeneration.

R. C. Glogau *et al.*, *Ind. Eng. Chem.*, 1961, **53** (4), 275-8.

Discrepancies between Design and Operation of Heat Transfer Equipment

This consists of a summary of papers presented at a panel discussion of the Heat Transfer Division of the American Institute of Chemical Engineers. Some questions and answers are also presented.

D. Q. Kern *et al.*, *C.E.P.*, 1961, **57** (1), 71-80.

Heavy-water Moderated Reactors Cooled with Light Steam-water Mixtures

Qualitative considerations on the general economy of heavy-water moderated reactors, fuelled with natural uranium, show the importance of the coolant. Cooling with light steam-water mixtures seems to be most promising because such a coolant approximates the ideal coolant. The paper reviews the problems arising in heat transfer, hydrodynamics, corrosion and erosion.

M. Silvestri, *Energia Nuc. (It.)*, 1961, **8** (3), 209-12.

Hydrogenations with Platinum Metal Catalysts

The paper reviews the hydrogenation of aromatics, nitro cpds, nitriles, oximes, carboxylic acids, ketones, aldehydes, olefins and acetylenes with platinum metal catalysts.

P. N. Rylander, *Englhrd Ind. Tech. Bull.*, 1960, **1** (3), 93-7.

Purification of Coke Oven Gases with Platinum Metal Catalysts

This paper reviews the catalytic removal of impurities such as NO, NO_2, C_2H_2, COS and diolefins from coke oven gases. 10 refs.

H. Andersen *et al.* *Englhrd Ind. Tech. Bull.*, 1960, **1** (3), 106-8.

Erythritol and Ethylene Glycol from Dialdehyde Starch

A process is described for making erythritol from dialdehyde starch for about $0.75 per lb. Present cost of erythritol is $200 per lb in small lots. 8 refs.

F. H. Otey, *Ind. Eng. Chem.*, 1961, **53** (4), 267-8.

Spherical Agglomeration of Solids in Liquid Suspension

The authors describe a new method for separating solid suspensions from liquids—spherical agglomeration. A basic requirement of the operation is the preferential wetting of the solid by a second liquid. Emulsions may be broken by adding a finely divided solid, the surface of which is repellent to the continuous phase and wetted by the emulsified phase. Some laboratory-scale experiments were carried out with a number of suspensions to illustrate the process.

J. R. Farnard *et al.*, *Can. J. Ch. E.*, 1961, **39** (2), 94-7.

Catalysts for Flameless Combustion

Chromium oxides, chromium chromates and chromium complexes are claimed to be active catalysts for flameless combustion of organic liquids or gases at relatively low temperatures. They will also remove organic contaminants from waste gases. Combustion of CO_2 and water occurs at about 300°C or over.

Brit. Pat. Spec. 862,771.

Troubleshooting New Processes

The article presents a broad summary of typical operating techniques that have been found generally useful in plant start-ups.

J. E. Troyan, *Chem. Eng.*, 1960, **67** (23), 223-6.

A New Concept of Cooling Tower Design

This paper describes a new type of cooling tower which uses specially treated corrugated paper fitting. The paper is treated to increase its wettability and wet strength, to render it bactericidal and to minimize salt deposition during operation. These new towers were based on the principle that when water flows over a wettable surface, in a cooling tower, the air-water contact is greatly improved and the pressure drop is reduced.

In these particular towers, the sheet material was 0.15 mm thick compared to 3-4 mm for conventional plates, the fill was much lighter, weighing 60 kg/m³ dry and 200 kg/m³ wet and the overall dimensions of the tower were reduced.

A good water distribution system has also been devised.

To be published in Vol. 54, *Proc. Inst. R.*

GROUP F — PROCESS CONTROL

Functional Analysis of Manufacturing Processes in Chemical Industry

A rational analysis of a production chart leads to planned maintenance and savings resulting from reduced losses, unplanned shut-downs, excess stocks, etc. The author considers the stages in the manufacture of nitric acid: (a) the overall reaction $NH_3 + H_2O$ to produce acid of maximum concentration; (b) the efficiency of the catalytic oxidation; (c) flue losses; (d) operation of oxidation powers; (e) absorption powers; (f) cooling units; and (g) condensers, in which the functional analysis of a flow shut can often produce significant improvements.

J. Cathala, *Génie Chim.*, 1961, **85** S (3), 77-84.

Analyse Plant Control by Transient Response?

The author shows, by means of an application to a heat exchanger, that a transient response method for determining controller settings is just as good as frequency response methods.

R. J. Fanning, *Pet. Ref.*, 1961, **40** (3), 145-8.

A Method for Isolating Corrosive ... Gases ... from Measuring Apparatus Under Vacuum

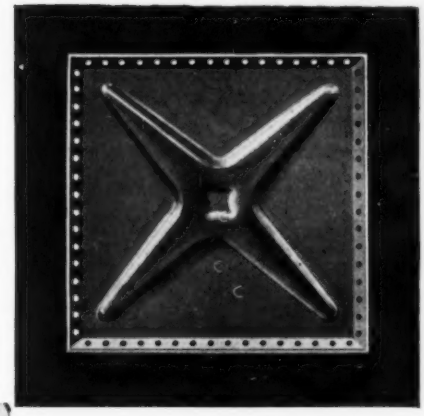
Vacuum measuring instruments or other apparatus can be protected from undesirable vapours by blanketing with a stream of a protective gas which is introduced through a jet. The author considers a suitable design of such an arrangement which is tested experimentally.

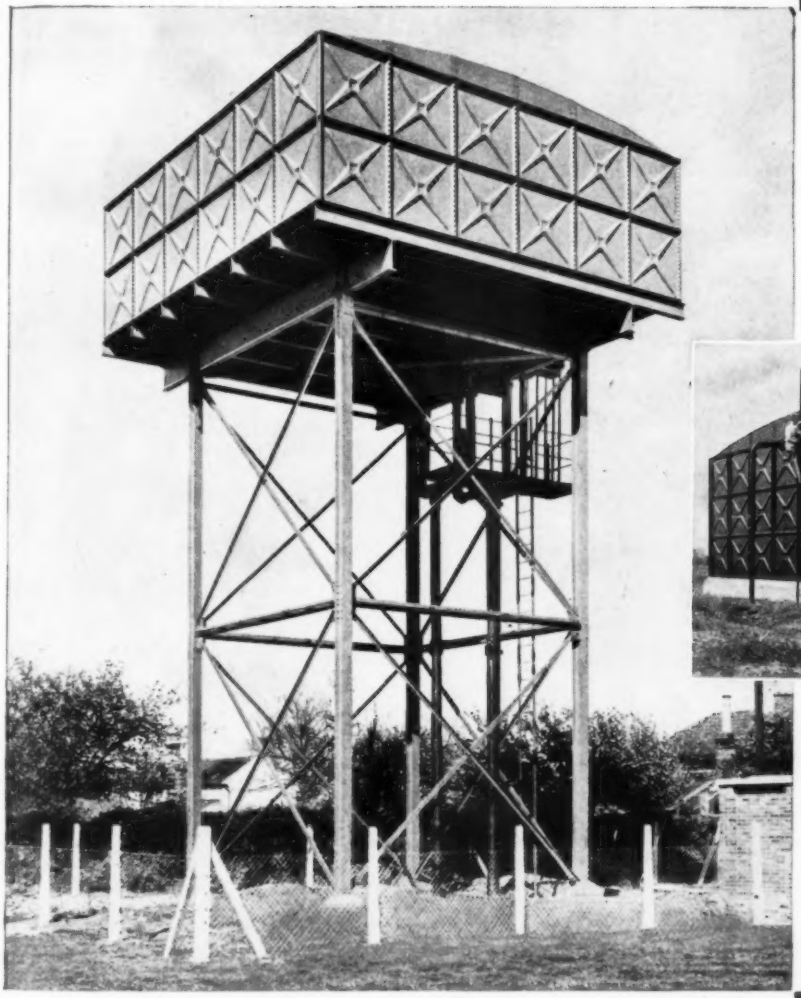
K. G. Muller, *Chemie Ing. Tech.*, 1961, **33** (4), 246-8.

Brit. Chem. Eng. Abstracts: **FOLIO No. 13**

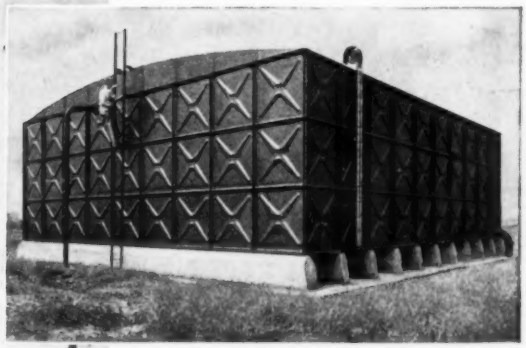
BCE 9784 for further information

BRAITHWAITE Pressed Steel Storage Tanks





Effective storage (from 220 gallons upwards) for water, heavy fuel oils and other liquids. These tanks, which can be enlarged or altered to meet varying needs, are light, strong and simple to erect with all tools supplied.



(left) 27,600 gallon covered tank 24′ square × 8′ deep with access ladders, supporting structure and valve control platform, supplied and erected at a housing estate.

(inset) Another covered tank for agricultural estate development 40′ × 30′ × 12′ deep capacity nearly 105,000 gallons.

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Abstracts

GROUP G — MISCELLANEOUS

Construction of the Reinforced Concrete Support for Rail-cranes at the Larymna Ironworks

The author describes the support, and its rather particular assembly with the steel rails. The whole system is subjected to heavy loads in a country (Greece) where earthquakes are frequent and severe.

G. Gross, *Génie Chim.*, **85** S (2), 65.

Size Reduction

A review of latest developments in size reduction which includes 173 references. A variety of crushing and grinding equipment is now designed on scientific principles.

R. V. Riley, *Chem. Proc. Eng.*, 1961, **42** (3), 117-23.

Differential Transmissions with Hydrodynamic Torque Converters

Having briefly explained the theoretical fundamentals of differential transmissions incorporating variable-ratio elements, the author deals in detail with the application of various formulæ to hydrodynamic systems. Characteristic features of hydrodynamic transmissions are evaluated and formulæ for calculating their parameters presented in tabular form.

C. Salamoun, *Strojírenství*, 1961, **11** (2), 106-15.

Application of Frequency Curves to Determining the Efficiency of Separators

An unusual method of determining the efficiency of separators from the frequency curves plotted for trapped dust is described. The method can be applied to the design of advanced separator for collecting extremely fine particles, even at very low concentrations.

Z. Svoboda, *Strojírenství*, 1961, **11** (2), 116-18.

Gaskets

The available literature in English, German and Russian languages has been surveyed and indications for correct design are given. Requirements for a satisfactory gasket are resistance to corrosion, erosion, mechanical shock and vibrations; it must also withstand the range of operating temperatures. Table 1 of the article offers guidance on the choice of jointing materials for different duties. After the correct material has been chosen, the dimensions of a gasket are obtained as follows: the critical precompression pressure (the minimum design seating stress) is determined together with the working pressure. The larger of the two is chosen and is checked to ensure that it is less than the maximum allowable gasket stress. The formulæ for designing both metallic and soft gaskets are given. When soft gasket material is used for gas installations there is some leakage through the pores of the jointing. If the physical properties of the gas are known the loss can be estimated from formulæ given; these are applicable to vacuum installations. 31 refs. (14 of them in English) are given.

G. Stvrteczky, *Magy. Kem. Lapja*, **16** (1), 38-44.

New Developments in Valves for High-pressure Plant

The paper describes the fabrication of considerably smaller and lighter valves for high-pressure plant, with self-sealing seals made from fluorocarbon plastics. Design and construction were influenced by requirement for good control.

The paper outlines the principles involved in these small valves and describes the seal and the U- and V-packing rings. Angle valves, membrane-controlled valves, poppet valves, non-return valves and branched pipes are described.

O. Konrad, *Chemie Ing. Tech.*, 1961, **33** (3), 195-204.

Aluminium—Material of Construction for Chemical Plant

This article discusses the use of aluminium and its alloys as materials of construction for chemical plant. Some of the equipment described is (a) tube in strip, for heat transfer, which is made from rolled sheet, delivered in coil or flat sheet and is then inflated pneumatically; (b) extruded finned tubes for heat exchangers; (c) roll bonded heat-transfer sheet; (d) cast valve bodies; (e) small pressure vessels; and (f) aluminium cladding material.

J. C. Bailey, *Chem. Proc. Eng.*, 1961, **42** (3), 137-43.

* Author's abstract.

How New Underwater Oil Storage Unit Works

The paper describes a self-contained, mobile, underwater oil storage unit of 20,000 brl capacity which can be sunk in 35 ft of water adjacent to a well platform.

It is designed for severe weather conditions and the steel storage tanks are fitted with cathodic protection and are protected from marine growth. The unit is suitable where a marine line to shore is uneconomical and offers adequate production facilities for deep water operations.

R. Lacey and J. Estes, *World Oil*, Nov. 1960, **151** (6), 92.

Handling and Transportation of Heavy Organic Chemicals

I. *General Considerations*

The paper stresses the advantages of bulk over packaged delivery and considers the types of bulk containers in current use as well as possible future designs. Two distribution problems are considered in order to illustrate the use of operational research.

C. D. Callieu, *Chem. & Ind.*, 1960, 1344-7.

II. *Technical Problems*

These include fire, explosion and toxic hazards, risk of contamination and choice of suitable construction materials for containers. Flexible containers (Dracones and Portolite tanks) and their uses are described.

J. W. Fletcher, Ibid., 1348-53.

Corrosion Design Problems in Chemical Process Equipment

Consultation between designers and corrosion engineers is necessary in planning chemical plant to minimize corrosion. Corrosion data sheets are presented and suggested as a means of obtaining information necessary to the corrosion engineer. Such data would not be more than is usually required for design specifications.

I. C. Bechtold, *Corrosion*, 1961, **17** (3), 14-15.

Influence of Acid and Chloride Concentrations on Corrosion in a Nitric Acid Concentrator

Heavy corrosion damage was caused by chlorides concentrated in parts of a large nitric acid concentrator from waste products in a uranium refinery. Chlorides were converted to chlorine by ozone, generated by electrical discharge, and by the use of electrolytic cells. The paper also presents extensive data on numerous corrosion-resistant metals in this environment.

J. B. Lowe, *Corrosion*, 1961, **17** (3), 26-8.

Fission Product Ruthenium in Effluent Treatments*

The categories of ruthenium complexes that exist in process solutions involving nitric acid have been studied in relation to their behaviour under certain typical floc precipitation conditions used in effluent treatment. It is concluded that nitrosyl ruthenium nitro complexes are primarily responsible for the poor ruthenium decontamination factors obtained in such processes.

E. R. Gardner and P. G. M. Brown, Report No. AERE R3551, Chemistry Division, U.K.A.E.A. Research Group, Harwell.

A Spouted Mixer-settler

The paper describes a new type of mixer-settler in which the heavy liquid phase is spouted upward through the light liquid phase and allowed to settle below it.

The mixer-settler is an effective continuous-flow unit which operates without the large energy losses inherent in mechanical agitation; mixing and settling occur continuously in the same chamber, a high-stage efficiency is obtained at optimum operating conditions and the design is simple. Its energy consumption is greater than that of an air-lift extractor.

Optimum conditions have been determined for bench-scale extraction of benzoic acid from toluene with a water spout. Murphree spouted-phase efficiencies up to 96 per cent were obtained. 20 refs.

T. R. Johnston, C. W. Robinson and N. Epstein, *Can. J. Ch. E.*, 1961, **31** (1), 1-8.

The Granulation of Carbon Black

The authors examine the formation of granules from one grade of carbon black in a small granulator. The void space was over 90 per cent and the presence of larger particles encouraged particle growth. Moisture content was the most significant single factor affecting the rate of granulation and strength of the granules. The optimum content was approximately equivalent to an adsorbed water layer. The role of this adsorbed layer in forming permanent bonds between particles is discussed.

T. K. Ross and T. Davies, *Trans. Inst. Ch. E.*, 1961, **39** (1), 28.

Brit. Chem. Eng. Abstracts: **FOLIO No. 14**

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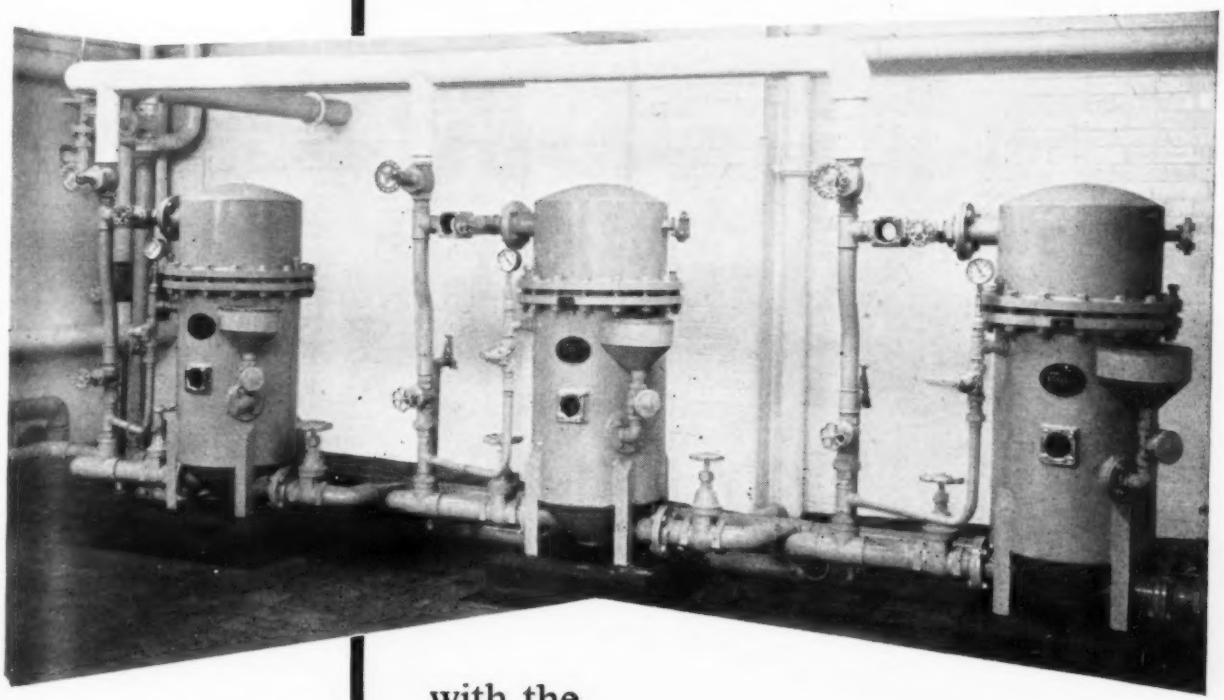

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Abstracts

GROUP G — MISCELLANEOUS (contd.)

Correlations in Heterogeneous Catalysis

A classification of types and classes of correlations in catalysis is proposed. Selectivity is more distinctive than activity as an index of catalytic action. Activity is, however, necessary in any catalytic system.

Correlations of catalytic activity are of two types: Type I, where a given reaction is studied on a series of catalysts and some kinetic parameter is related to a property of the solid catalyst or of a complex between catalysts and reactants; Type II, where a series of related reactions are investigated on a given catalyst and some kinetic parameter is related to a property of the reacting molecules. Correlations of Type I are more abundant than Type II.

In this review correlations of both types are presented to elucidate the characteristics of the catalysts (Type I) and the chemistry of catalysis itself (Type II).

M. BOUDART. Paper presented at the A.I.Ch.E. meeting in New Orleans, February 1961.

Pressure Losses in Dry Distillation Trays

Nine distillation trays were tested to determine the relationship between the pressure losses and the throughput rate in a research installation developed specially for studying pressure and hydraulic conditions in dry towers. The trays were six slotted trays, two modified slotted trays and a screen-type tray with circular holes.

M. SELIN, *Strojirenstvi*, 1961, **11** (4), 255-60.

Ireland's Chemical Industry

A recent tour of several representative branches of Ireland's chemical industry has convinced the author that, although small at present, this particular industry shows promise of considerable expansion during the next few years.

I. L. HEPNER, *C.P.E.*, 1961, **42** (4), 178-81.

Materials of Construction for Chemical Plant: Reinforced Plastics

The paper describes the various types of plastic reinforcement agents, glass, glass-fibre and resin; the properties of these materials of construction and their uses.

K. PARVIN, *C.P.E.*, 1961, **42** (4), 171-4.

Indication Diagrammes of High-pressure Gas Compressors

The paper describes a high-pressure gas compressor and methods of obtaining indicator diagrammes with three types of detector. The design of these detectors and their operation is described. An indicator diagramme obtained with an ionic glow discharge detector is given.

Translated from Russian. Available from office of Technical Services, U.S. Department of Commerce, Washington, 25, D.C. Paper JPRS 5526.

How to Evaluate Chemical Projects

A method is presented for selecting the best project. The method considers the costs, revenues and timing factor and is illustrated with an example.

H. C. THORNE, JR., *et al.*, *Pet. Ref.*, 1961, **40** (3), 171-6.

Nomographs Speed Vessel Design

Six nomographs are presented to assist the designer in solving rapidly the design formulæ in the 1959 A.S.M.E. Boiler and Pressure Vessel Code, Section VIII.

These nomographs give the thickness of cylindrical and spherical shells; ellipsoidal, hemispherical and torispherical heads; and maximum allowable working pressures.

F. CAPLAN, *Pet. Ref.*, 1961, **40** (3), 161-2.

Find Equilibrium Constants Graphically

This paper presents a rapid method for estimating equilibrium constants. Nomograms are presented which relate the chemical equilibrium constant K at any temperature to the heat and free energy changes for the reaction in the standard state at 298°K. The nomograms can be used to check the thermodynamic flexibility of a reaction, to determine the temperature required for a certain conversion, and to estimate the change of phase equilibrium constants with temperature.

E. A. DESTREMPS *et al.*, *Pet. Ref.*, 1961, **40** (3), 163-8.

* Author's abstract

Estimating Air-cooled Exchangers

A rapid and simple method for estimating the approximate size and horsepower requirements for air-cooled heat exchangers with standard-type fin tubes is described.

J. W. MURTHA and S. H. FRIEDMAN, *Chem. Eng.*, 1961, **68** (3), 99-100.

How Statistics Apply to Chemical Processes

Application of new statistical techniques can result in significant economic advantages.

The author describes the development and present status of statistics, as applied to chemical processing, and focuses attention upon those techniques that have proved their value in process development and improvement.

T. L. KOEHLER, *Chem. Eng.*, 1960, **67** (25), 142-52.

Improving Boiler Efficiency

The paper shows how savings in fuel gas consumption of boilers, 30 or more years old, can be effected by reducing the excess air. A chart and a formula are given to find the excess air, if either the CO_2 or the O_2 content of flue gas is known.

E. MASON, *Pet. Ref.*, 1961, **40** (4), 117-20.

The Effects of Nuclear Radiation on Thermocouples

The formation of new metals resulting from the transmutation of elements caused by neutron capture may, over a period of time, cause incorrect readings from thermocouples used in nuclear experiments. The amount of new metal formed depends on the neutron flux and on the ease with which an atom can capture a neutron and become a chemically different atom.

With the exception of rhodium, common thermocouple materials have a poor ability to capture neutrons. Previous experimental work has been little affected by this phenomenon. However, extremely high-temperature research of the present and future requires refractory metal thermocouples, and their ability to capture neutrons is higher than that of conventional materials.

M. J. KELLY *et al.* Paper B-3-3 presented at the 1961 Symposium on Temperature, sponsored by Am. Inst. of Physics, Nat. Bur. of Standards and Instrument Soc. of Am.

Thermocouples for Measurement of Transient Surface-Temperatures

This paper describes a type of thermocouple used for studying reactions that occur in extremely short periods of time. Some examples of its use are (a) indicating the internal surface temperature of a machine-gun barrel when a bullet is fired, (b) to study how various fuel build-up deposits on the internal surfaces of gasoline engines.

C. E. MUELLER. Paper B-4-3 presented at the 1961 Symposium on Temperature, sponsored by Am. Inst. of Physics, Nat. Bur. of Standards and Instrument Soc. of Am.

Chemical Rocket-propulsion Systems

The paper presents an account on the present state of the chemical rocket-propulsion field with a critical examination of liquid and solid propellants. 37 refs.

F. J. HENDEL, *Chem. Eng.*, 1961, **68** (5), 99-114.

The Cause of Piping Vibrations*

Pipeline vibrations are classified as free, forced or self-excited. Various examples of pipeline variations and their effects are given and methods are suggested for removing the vibrations.

J. P. CHAMBERLAIN, *Pet. Eng.*, 1960, **32** (10), C30.

A Statistical Approach to the Estimation of Design Data

This report is written for design engineers and others who are faced with the task of making safe estimates from the evidence of one or more tests. The method is based on sampling theories of statistical analysis and makes use of confidence limits, exclusion limits, coefficient of variation and Test Acceptance Factor. Three examples of its application are given.

A. N. KNOWLES, U.K.A.E.A., Risley, Lancs. 1961 DEG Report 316 (R). Obtainable from H.M.S.O. 3s. net.

Pressure Vessel Design for High or Cryogenic Temperatures

This article contains charts of tangential and longitudinal stresses vs t/R for pressures ranging from 250 to 3000 psi; where t = vessel thickness, R = radius of shell; and charts giving thermal stresses in the t/R range 0.1 to 1.0.

These charts will assist in the design of vessels in accordance with the A.S.M.E. Unfired Pressure Vessel Code.

Two examples are given: one for high pressure (1250 psi) and high temperature (950°F); the other for high pressure (3000 psi) and low temperature (−250°F).

R. R. MACCARY, *Chem. Eng.* 1960, **67** (24), 131-6.

Brit. Chem. Eng. Abstracts: **FOLIO No. 15**

BCE 9786 for further information

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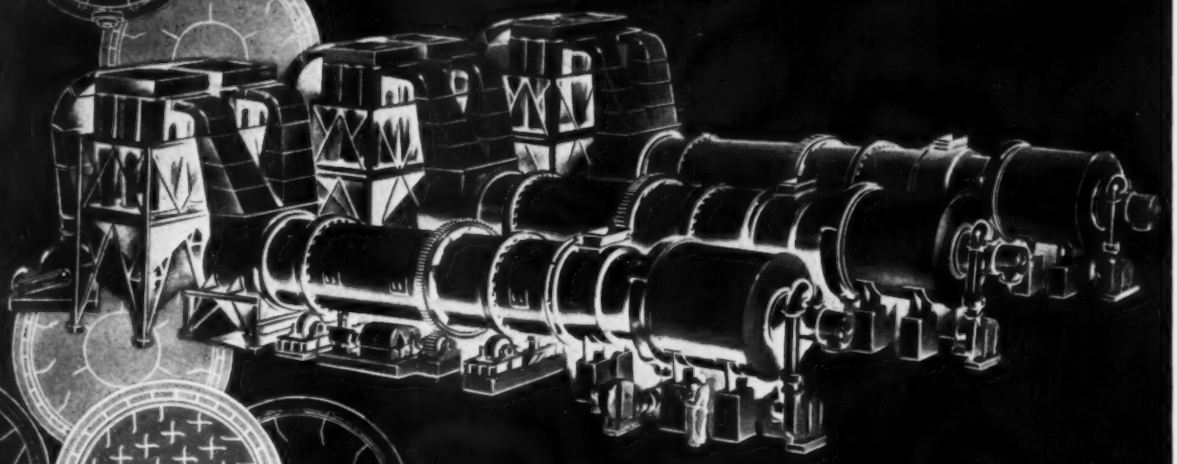

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Abstracts

GROUP G — MISCELLANEOUS (contd.)

Selecting Materials for Process Piping

A report on this subject covers a wide variety of piping materials: ferrous alloys, aluminium, lead and silicon irons, graphite, plastics.

C. K. ALDRICH, *Chem. Eng.*, 1960, **67** (23), 183-92.

Oxidation-reduction Catalysis

The paper deals with the possible use of chromites to reduce air pollution from exhaust fumes by removing nitrogen oxides, hydrocarbons and CO.

J. F. ROTH and R. C. DOERR, *Ind. Eng. Chem.*, 1961, **53** (4), 293-6.

Ageing of Platinum-reforming Catalysts

This paper discusses the regeneration of platinum-reforming catalyst by reversing the reactions which cause ageing of the catalyst.

C. G. MYERS *et al.*, *Ind. Eng. Chem.*, 1961, **53** (4), 299-302.

An Improved Gasoline Anti-detonant

A study of the mechanism that makes TML a more effective anti-knock agent than TEL in high-octane gasolines.

W. L. RICHARDSON *et al.*, *Ind. Eng. Chem.*, 1961, **53** (4), 305. Also *Journal of Chemical and Engineering Data*, April 1961, Vol. 6.

Double Pipe Heat Exchangers

A method is described for obtaining the heat exchange area required when a liquid is to be heated by a condensing vapour. An example is given for water heated with steam; the result is 1.7 per cent lower than the value obtained by graphical integration.

S. L. SULLIVAN, JR., and C. D. HOLLAND, *Ind. Eng. Chem.*, 1961, **53** (4), 285-8.

Corrosion in Cryogenic liquids

An examination of titanium and its alloys at low temperatures (−300°F to −350°F) and in contact with liquid oxygen and liquid fluorine. 10 refs.

J. D. JACKSON, *C.E.P.*, 1961, **57** (4), 61-4.

New Alloys for Extreme Conditions

New metals and alloys are being developed to meet the severe requirements of present-day technology. This paper reviews nickel alloy steels and other alloys at liquid gas temperatures ranging from −43.7 to −452.1°F. 13 refs.

E. G. HOLMBERG, *C.E.P.*, 1961, **57** (4), 74-8.

Catalytic Reactor Design

The authors combine reaction kinetics and a gas flow model to develop a method for scale-up of fluid bed data, for the oxidation of naphthalene to phthalic anhydride.

F. DE MARIA *et al.*, *Ind. Eng. Chem.*, 1961, **53** (4), 259-66.

Some Rheological Parameters of Clays and their Thixotropic Behaviour

Four clays, Kaolinite, Illite, Montmorillonite and Attapulgite, were studied. Clay-water samples were prepared at about their liquid limits.

The yield value and viscosity coefficient were obtained from stress to strain relationships. Regain of strength was evaluated in terms of viscosity and yield values at various periods of ageing. The influence of temperature on ageing was determined for attapulgite.

The activation energy of ageing, calculated for attapulgite, was 9.4 Kcal/mole, which suggests a physical change.

A. S. YALCIN and R. MCINTOSH, *Can. J. Ch. E.*, 1961, **39** (2), 76-80.

Anti-vibration Mountings

This comprehensive survey covers a great variety of anti-vibration mountings.

Engineers' Digest, 1961, **22** (4), 77-102.

Optimum Process Conditions for a Completely Mixed Multi-stage Reactor

The authors present a graphical method for determining the process conditions which give the minimum volume of reactor for a given outlet conversion of a specified reactant. The method is applied to a three-stage fluidized-bed reactor for the catalytic oxidation of SO_2.

H. KUBOTA *et al.*, *Can. J. Ch. E.*, 1961, **39** (2), 64-6.

* Author's abstract.

Blast-furnace Slab as an Aggregate in Reinforced Concrete*

The role of blast-furnace slab as an aggregate in reinforced concrete is considered both in the light of present-day experience and in past erroneous reports. It was found that such slag is substantially inert in so far as any deleterious action towards steel is concerned. Several pre-stressed concrete structures containing slag aggregate, in use for more than three years, showed evidence of giving long and trouble-free service.

C. P. LARRABIE and S. K. COBURN, *Corrosion*, 1961, **17** (4), 79-80.

Increasing the Capacity of Nitric Acid Plants by the Use of Commercial Oxygen

This investigation of the use of commercial oxygen revealed the following. (1) Partial or total substitution of the air by oxygen added to the denitrating tower will increase the amount of ammonia which can be processed. (2) When the transfer capacity of the turbo-compressor is fully used, the amount of ammonia will be determined by the sum of infiltrated air and the air used for denitration. (3) When less than the maximum amount of ammonia is processed, partial or complete substitution of denitrating air by oxygen increases efficiency through reduced loss of nitrogen oxides. (4) The increase in output of the plant will be economical only if the oxygen is manufactured on a scale greater than demanded by this purpose alone. (5) The capacity may be increased more economically by allowing a greater loss of nitrogen oxides than by manufacturing oxygen especially for this purpose.

B. ENYEDI, *Magyar Kemikusok Lapja*, Vol. 15, 1960, 5-6, pp. 244-51.

The Manufacture of Ethylene Oxide

The author compares two processes which are used, almost exclusively, for the production of ethylene oxide: the chlorhydrin process and the direct oxidation process. A distinct preference is apparent for the direct oxidation process mainly because it does not give rise to by-products, does not require chlorine, and has been simplified due to chemical engineering developments since the war.

R. P. VAN OOSTEN, *J. Inst. Pet.*, 1960, **46**, 347-51.

Industrial Chemicals Based on Ethylene and Propylene

The paper surveys the wide field of chemicals produced from these two olefins. New processes and reactions are being continually developed, recent examples being: the oxidation of iso-propanol to acetone and H_2O_2, the oxidation of ethylene to acetaldehyde and the reaction of propylene with NH_3 and air to give acrylonitrile.

A. F. MILLIDGE, *J. Inst. Pet.*, 1960, **46**, 353-66.

Symposium on Particle Fluid Interactions

This 93-page volume in three sections contains thirteen papers on several aspects of particle fluid interactions. I. Five papers on research associated with current reactor programmes. II. Three papers on fluidized-bed studies related to nuclear technology. III. Five papers on various basic studies related to fluidized beds, turbulent suspensions, viscous flow around particles and fluid turbulence.

Held at New York, May 1959. U.S. Atomic Energy Commission, Office of Technical Information: TID-7592.

Heat Transfer

This 208-page volume contains twenty papers selected from those presented at the Fourth National Heat Transfer Conference held in Buffalo, August 1960, and three papers selected from recent short symposia. This issue reflects activity in the fields of heat transfer during two-phase flow, heat transfer during phase changes, and heat transfer in packed or fluidized beds.

Chemical Engineering Progress Symposium Series, 1961, **57** (32), published by American Institute of Chemical Engineers.

Apparatus for Study of the Break-up of Liquid Drops by High-velocity Airstreams

The energies required to atomize liquids are very large, and high-energy methods are required to study this high-energy process. A blast gun and shock tube were used to investigate the break-up of liquid drops, 3-4 mm dia, in high-velocity air streams. Flow in the shock tube was uniform and predictable; pressures and velocities behind the shock front could be regulated as desired. Preliminary observations were made at approx. Mach 1 airstream velocity, and 1 atm pressure behind the shock front. Photographs show a gradual transition in break-up mechanism as the forces acting on the drop, caused by the high-velocity air stream, are increased. 12 refs.

J. D. WILCOX and R. K. JUNE, *J. Franklin Inst.*, 1961, **271** (3), 169-82.

Brit. Chem. Eng. Abstracts: **FOLIO No. 16**

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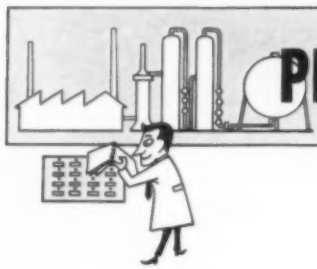
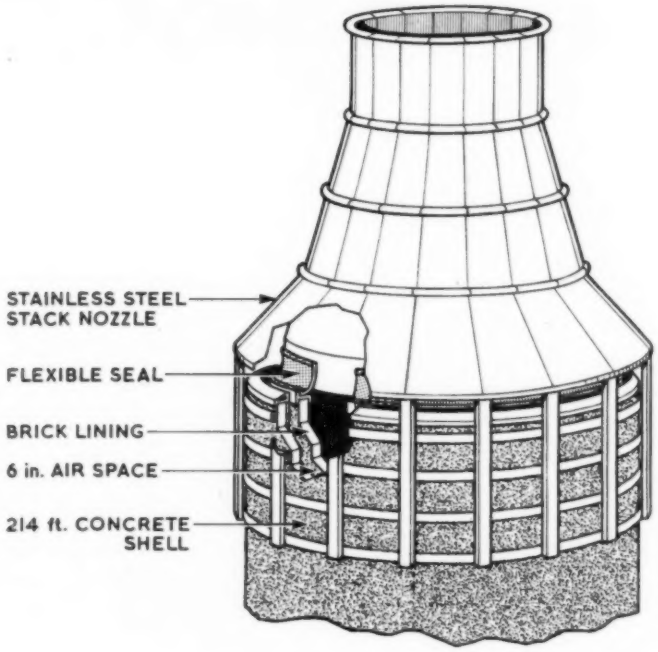


PROCESS ENGINEER'S NOTEBOOK

Increasing Effective Height of a Stack

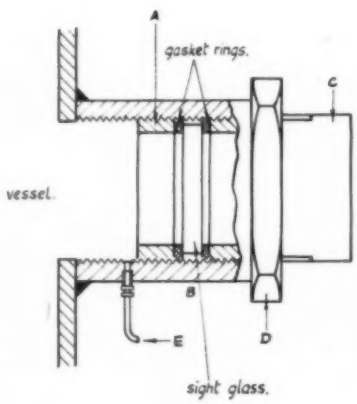


A METHOD of increasing the effective height of a stack to obtain better effluent dispersal consisted of adding a specially fabricated nozzle at the top, the idea being to increase the velocity of the exhaust gases sufficiently to correspond to the desired increase in effective height. This approach was used by the Aerotec Industries Inc., of Norwalk, Conn., U.S.A., who incorporated in their nozzle a double shell skirt. Design of the nozzle was complicated by the dual brick-concrete construction of the chimney. The 19 ft o.d. outer concrete shell is separated from the inner brick lining by a 6-in. air space. This clearance permits the chimney to sway under wind loads while the brick lining, except for thermal expansion, remains stationary. The problem was to attach the nozzle to the inner, gas-bearing brick liner without obstructing the annular 6-in. sway-clearance space between it and the concrete shell.

The inner skirt was attached to the brick liner by a flexible diaphragm, while the outer skirt was attached to external supports on the concrete stack. The total weight of the heavy load was cantilevered from the load-bearing concrete, its inner surface being mated with the brick liner so that there was no interference with the space between the two. The flexible diaphragm was made from PTFE fibre impregnated with a fluoro elastomer. This seal was completely flexible, had ample mechanical strength and resisted the action of the corrosive gases, which were at a temperature of 200°C. The sole penalty for the addition of the nozzle to the stack was an increase of only 2 in. water gauge in the resistance to flow. The nozzle is able to project 485,000 cfm of gas more than 100 ft above the chimney top.

A Temporary Sight Glass Arrangement

IN a process using flowing solids certain irregularities in flow and behaviour occurred which made it desirable to observe visually the inside of the reaction vessel. Fortunately, standard 1½-in. sockets had been welded to the outer wall of the vessel during its construction for probe insertion: the sockets made it possible to install quickly a simple sight glass system. The diagram shows the method used.



A short length of standard 1½-in. "running" nipple (A) was screwed into the socket (B) to a distance of about 1 in. to form a backing rim for the sight glass. Glass discs of slightly less diameter than that of the socket bore were available, but in any case are easily obtainable. To form a seal it was necessary to cut two soft asbestos washers and stick one to each side of the sight glass (using a proprietary cementing material) before pushing it into position against the backing rim (A). The glass, together with its washers, was then tightened against the backing rim by screwing in another 1½-in. nipple (C), which was provided with a locknut (D). The locknut (D) saved the glass from damage by overtightening and held the nipple firmly in the socket. A further measure, which was found to be necessary because of the dustiness of the material in the reaction vessel, was to provide for a small gas bleed (E) on the inside of the sight glass to keep the socket clear of accumulating dust.

This type of sight glass arrangement has been used successfully in plant handling solids at 800°C. The "sight glass" itself used in this case was a commercially available silica disc to withstand the temperature.

A Flange Arrangement for Vessels Handling High-velocity Solids

APART from general abrasive wear of the walls of vessels handling solids moving at high velocity (e.g., as in cyclone separators), an acute "joint scouring" problem can arise in vessels which are made up from separate sections. The established procedure for joining vessel sections together is to use the bolted flange method of connection and to incorporate some form of gasket ring between the flanges. Normally the gasket itself would be exposed to the conditions within the vessel and, in most applications, this would not matter. However, there are certain operations involving high velocity solids at high temperature, when the conventional method of flanging would be unsatisfactory and could be dangerous.

In a small pilot plant handling hot solids at high velocity, some warping occurred in flanges connecting two sections of a vessel. Continual abrasion of the gasket material by the hot solid eventually disintegrated it and caused a dangerous leakage of red-hot material.

The problem was solved in later vessels of the same type by "offset fixing" of the flanges as shown in the sectional diagram. Fig. (a) shows a normal flange connection; Fig. (b) shows the method adopted to prevent scouring of the joint.

One flange (A) was welded above the wall end; the mating flange (B) was welded a short distance up its own wall, so leaving a lip of wall to protect the joint ring.

Since this method of flange connection was adopted, there have been no further instances of joint scouring and leakage.

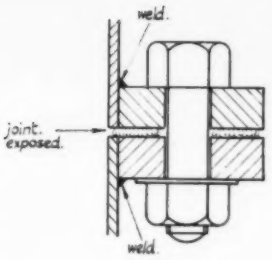


FIG. (a)

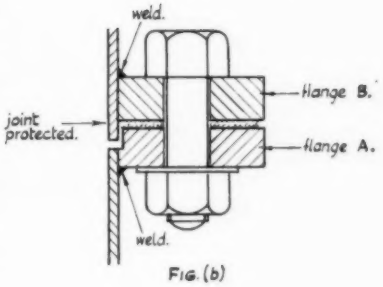


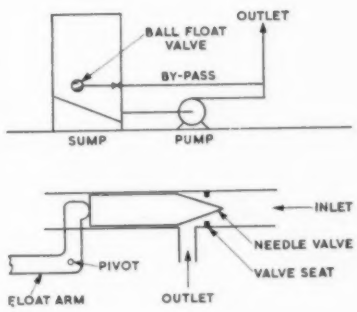
FIG. (b)

Overcoming Variable Feed to Pump

THE method of controlling a centrifugal pump, described on page 298 of the May issue of this journal, has the disadvantage that air is drawn into the pump and hence into the system. A simple method of overcoming this is to fit a by-pass between the output of the pump and the sump, and to control the flow of liquid in this by-pass by a domestic ball float valve in the sump (Fig. 1). Thus when the sump liquid level falls the by-pass opens and the pump output is reduced. As the liquid level in the sump rises the by-pass closes and the pump once again runs at full capacity.

With the normal type of valve this control will tend to be "on-off", as a small movement of the ball float valve will fully open or close the valve. However, the valve may easily be adapted (Fig. 2) so that it has a proportional throttling action, and it will then require a large movement of the float to move the valve from fully open to fully closed. This not only prevents air from being drawn into the system, but very effectively reduces the fluctuations in the flow.

A further advantage of this system is that, if the feed rate to the sump is constant but less than the pump capacity, the pump will not work intermittently, but will give a steady output, equal to the feed, while returning a proportion of the liquid through the by-pass. J.R.C.

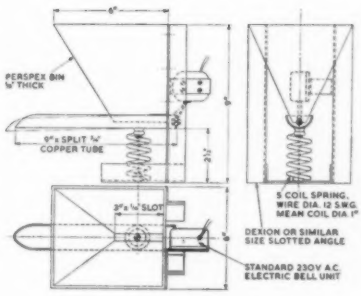


Pilot Plant Continuous Solids Feeder

CERTAIN pilot-plant experiments on the continuous extraction of soyabean oil from soyabean meal required a small continuous solids feeder to be made at short notice. Accordingly, a design of vibrator feeder was decided upon. This was made up from odd material available at the time and was found to perform so well on this duty that it was felt that the design might be useful in certain industrial operations, such as continuous blending of various inorganic and organic pigments, etc.

The amplitude of vibration can be readily varied to provide a certain flexibility in the capacity of this particular feeder.

F. MOLYNEUX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Contributions Invited

The Editor invites contributions to "Process Engineer's Notebook". They should preferably deal with practical devices designed to overcome problems for which standardized equipment is not available; hints for the improved design of conventional equipment; methods of improving equipment operation; safety devices; etc. For the most meritorious contribution the sum of **10 gns.** will be paid, while other contributors will receive **5 gns.** They will be published anonymously or under the contributor's name, according to his wishes.

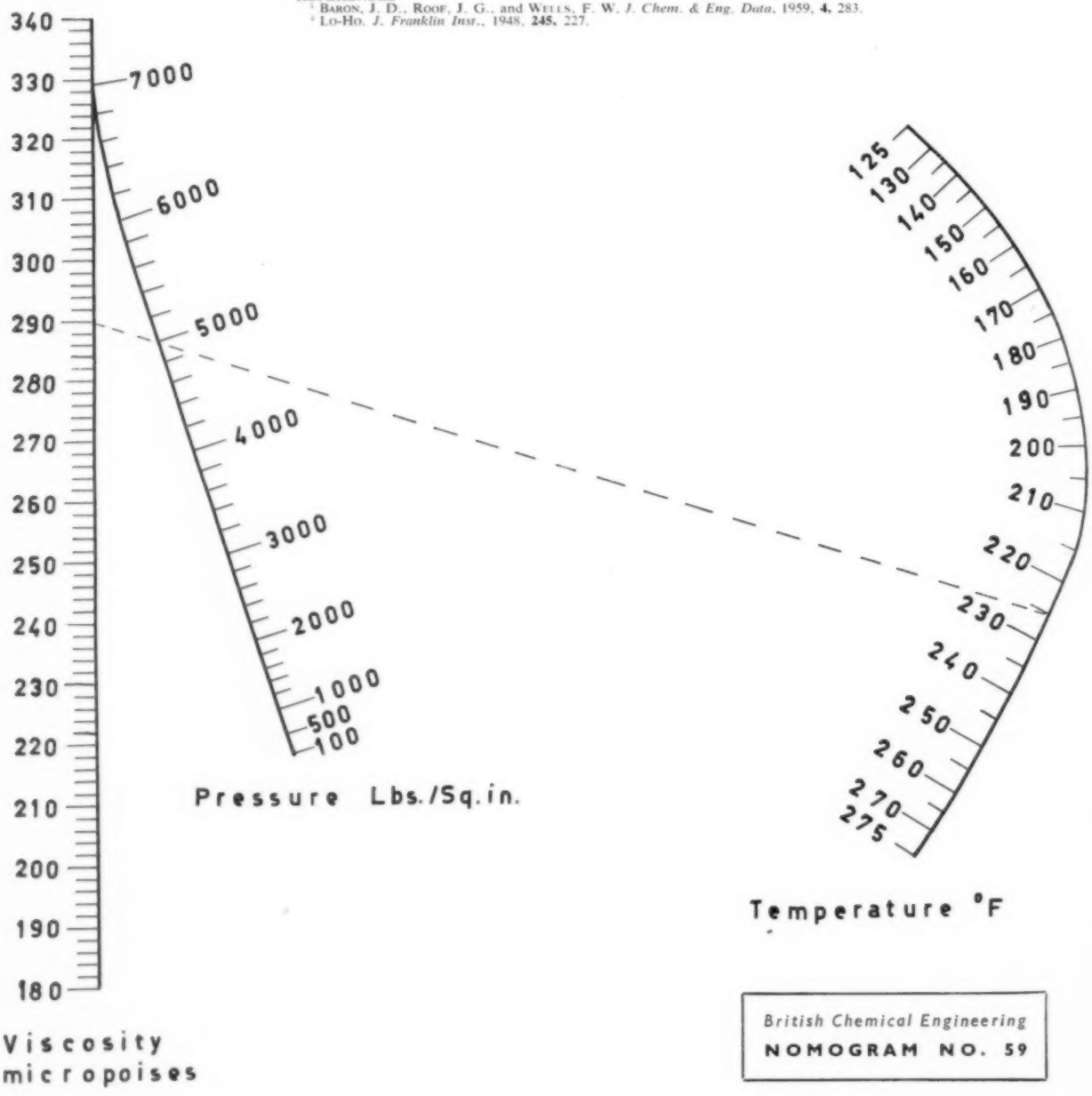
VISCOSITY OF NITROGEN

by **A. M. P. TANS**
(Central Laboratory, Staatsmijnen in Limburg, Geleen, the Netherlands)

Recently BARON, ROOF and WELLS[1] measured the viscosity of nitrogen as a function of temperature and pressure. From these measurements the accompanying nomogram has been constructed.[2] The largest deviation amounts to 1.5 micropoises.

The dotted line indicates that at 225°F, and at a pressure of 5000 psi, nitrogen has a viscosity of 289.5 micropoises.

REFERENCES
[1] BARON, J. D., ROOF, J. G., and WELLS, F. W. *J. Chem. & Eng. Data*, 1959, **4**, 283.
[2] LO-HO. *J. Franklin Inst.*, 1948, **245**, 227.



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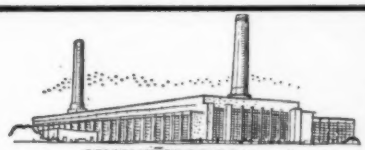

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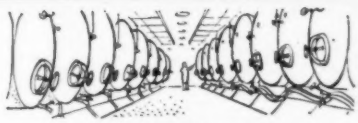

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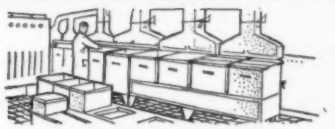

P.V.C. *fabrication scheme for fume extraction*

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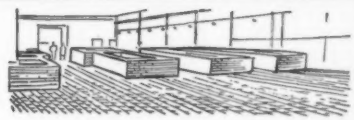

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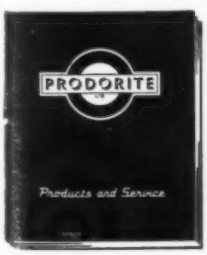

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Book Reviews

Fluidization and Fluid Particle Systems
by F. A. Zenz and D. F. Othmer
Reinhold, 1960, 120s. (Agents in U.K., Chapman & Hall.)

THIS book makes reference to a great number of publications related to fluidization and bordering fields. This will make it indeed valuable to those desiring an overall view of what has been done in this region.

The book starts with a description of existing processes that use fluidization in one form or another. The following sections on properties and gravity flow of powders, previously published in the *Petroleum Refiner*, make interesting reading, though it is felt that this coverage by some 100 pages may perhaps be slightly excessive, especially when compared with the somewhat hurried discussions of other fields, notably mass transfer, to which only 13 pages near the end are devoted.

Flow of single-phase fluids through fixed beds is reviewed in some detail. Curiously, and perhaps somewhat out of place, there appears one brief reference to so-called "Trickle-Flow", which is to denote gas-liquid flow through packings, a subject on which a very large and by no means non-controversial literature exists by itself. The discussion of flow through the fluidized bed follows along the line of the treatment of WILHELM and KWAUK for particulate fluidization. A possible extension of this to aggregative systems is suggested, but may, however, be open to question. In subsequent chapters the bubble phenomena are well covered and a new approach for estimating carry-over from fluidized beds is outlined in much detail along the lines of an earlier journal article by ZENZ and WEIL. Though interestingly presented, the reviewer feels that the presentation could have been elucidated greatly by applying all this to a typical problem. This is always helpful, as it guides the unfamiliar through any labyrinth which a new approach may be so often to so many.

Flow of solids and pressure drop in pneumatic systems is also well covered and in the chapter on heat transfer a fairly large number of the more important correlations have been presented. Regrettably, though, some key studies seem however to have been treated far too briefly, as for instance the important contribution of VAN HEERDEN and co-workers, which are barely mentioned. Similar omissions are noted in other areas. Thus, for example, the fundamental expansion studies of RICHARDSON and ZAKI are not covered anywhere, as in general, in the opinion of the reviewer, not sufficient coverage is accorded the efforts documented by the Association Francaise de Fluidisation and more recent developments in Canada on the subject of spouting. It might also have been well at this time to attempt a co-ordination of some kinetics considerations with fluidization characteristics. Despite these specific omissions, however, the tremendous effort of screening the large segment of literature covered must be truly appreciated, even though coverage was perhaps not as critical as might have been desirable.

The style of presentation is fluent and the reader is kept interested at all times. The entire nomenclature is presented at the end of the book and symbols are thoughtfully chosen, and the book, well done in its execution, appears to be quite free of errors. This most recent comprehensive writing on fluidization should be a most welcome source and reference book to many.

MAX LEVA

Molecular Distillation
by G. Burrows
Oxford University Press, 1960, 214 pp., 35s.

THIS book is the latest member of the Oxford series of monographs on the Physics and Chemistry of Materials, and its author, who is in the research department of Associated Electrical Industries Ltd., is one of the foremost authorities in the field of molecular distillation.

One might perhaps have expected this volume to be well studded with esoteric pieces of physical chemistry enlivened by brief details of laboratory apparatus, the whole effect being so learned as to put off all but the earnest Ph.D. student. Instead, we find a neat and adequate introduction to the theory of the process, but the major part of the text concerned with practice and design both on the laboratory and plant scale.

Basic theory is dealt with in the first two chapters, with some overflow into Chapter 3, which is entitled "The Representation of Results". Vacuum technique mechanical design and physical design are covered in the next three chapters, and then the foregoing material is combined and illustrated by examples of apparatus and details of operation and control.

The text proper ends with an excellent survey of the applications of the process, past, present and future. A useful appendix—ranging from data on pressure equivalents to a formula for calculating the number of molecules in a monomolecular layer—and a list of references complete the book.

The chemical engineer will find the treatment of distillation theory somewhat novel and stimulating. One would have imagined that the well-worked field of graphical representation of distillation processes had been cropped to exhaustion, but a new one is harvested here.

It is a very well written and easily readable book with nicely balanced contents containing something for the chemist, the physicist and the engineer. It will certainly improve any technologist's bookshelf and is a "must" for anyone concerned with molecular distillation, be he designer or user.

D.C.F.

Small Particle Statistics
by G. Herdan
Revised and enlarged second edition
Butterworths Scientific Publications, 1960

THE object of the first edition of HERDAN'S book (1953) was to establish the statistics of particles in the sieve and sub-sieve range as a branch of the discipline of statistics in its own right. One cannot say today that this object has been fulfilled. However, the book has always had a much wider appeal.

First the author has made a unique and valuable contribution to the analysis of data on the size of single or aggregates of particles by elaborating on the statistical techniques which physicists and engineers should use when handling such data. As such, it is a book which requires some preliminary understanding of elementary statistical methodology before it can be fully appreciated.

Secondly, the book deals extensively with methods used for size determination and not only with the statistics of particles. One would have hoped that this new edition would have provided an opportunity to change the title and to make the cumbersome arrangement of the book more attractive as distinct from the type and setting which has indeed been greatly improved.

The word "statistic" is just not suitable to describe the contents of the book, neither is it appealing to the majority of particle technologists who, by not being able to overcome the intellectual hurdle which is presented by such a title and by many subtitles, miss the many treasures of information which are offered. What is one to make of the title of Chap. 12: "Physical and chemical properties of materials in their dependence upon characteristics of the materials as particulate universes"? Another instance is the last chapter of the book entitled "Experimental design and experimental errors of particle size determination", which had originally been written by M. L. SMITH who is joined in this edition by W. H. HARDWICK and P. CONNOR, all of the A.E.R.E., Harwell. It contains descriptions of the various experimental methods for determining size distribution and surface area and only differs from the rest of the book by almost omitting any mention of statistics, or experimental error or design. Since analysis and determination of the statistics are closely related, these nine chapters could with advantage have been spread over the rest of the book.

The 50-page enlargement of the book constitutes a valuable addition. It consists on the whole of experimental descriptive material on centrifugal sedimentation, radiometric techniques, airborne particulate matter, automatic counting and sizing equipment, and some additional statistical techniques on the analysis of variance, measures of the degree of mixing and the fitting of frequency distributions.

One may hope that this book will sell so well or that other means can be found to bring out a third completely revised edition.

A.B.C.

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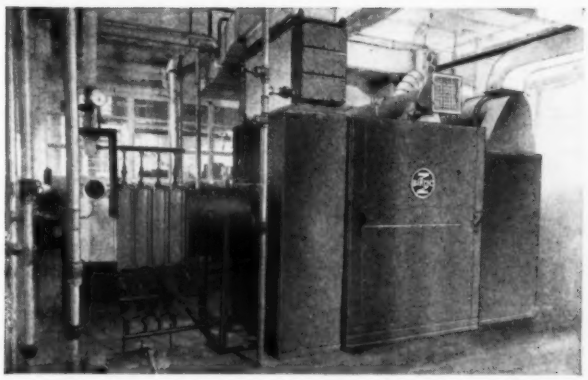

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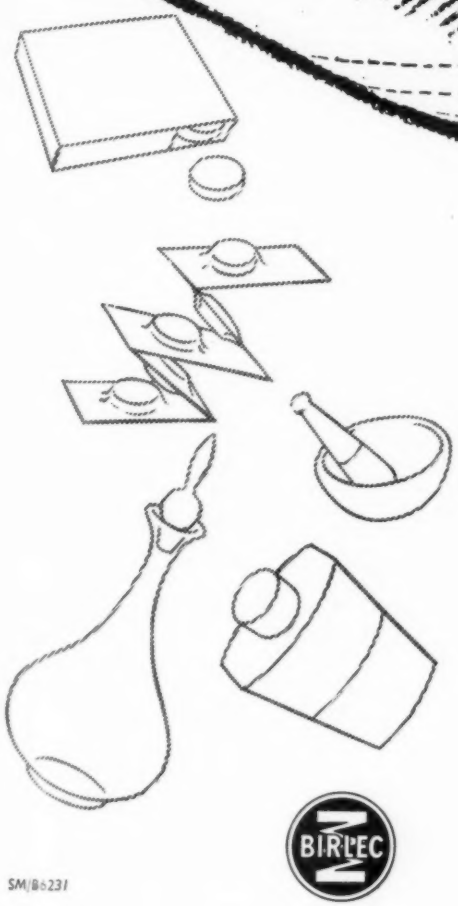

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PLANT EQUIPMENT NEWS

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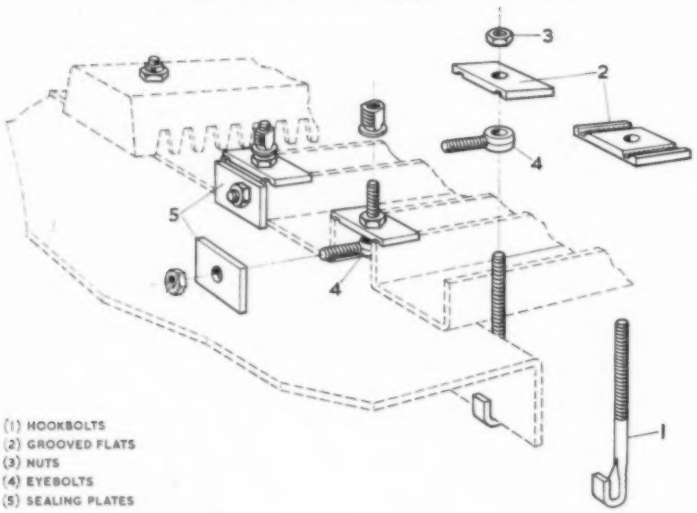

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BCE 9838 for further information

Conveyor Safety Device

This is an emergency-stop device for conveyors which is operated by a beam of light and a photo-electric cell that is immediately effective whenever the operator's hand is passed over the conveyor at any point between its ends.

The device cuts to an absolute minimum the time between an emergency being detected by the operator and stoppage of the conveyor, thus increasing operator and plant safety. The unit has also been arranged to fail safe so that component failure will stop the conveyor. The device is simple to install and in single units can be used with ray lengths of up to 75 ft. The equipment can be supplied in flameproof certified housings. Prices vary, according to the length of ray, from £20 to £30. **Hird-Brown Ltd., 244 Marsland Road, Sale, Cheshire.**

BCE 9839 for further information

Pneumatic Transmitter

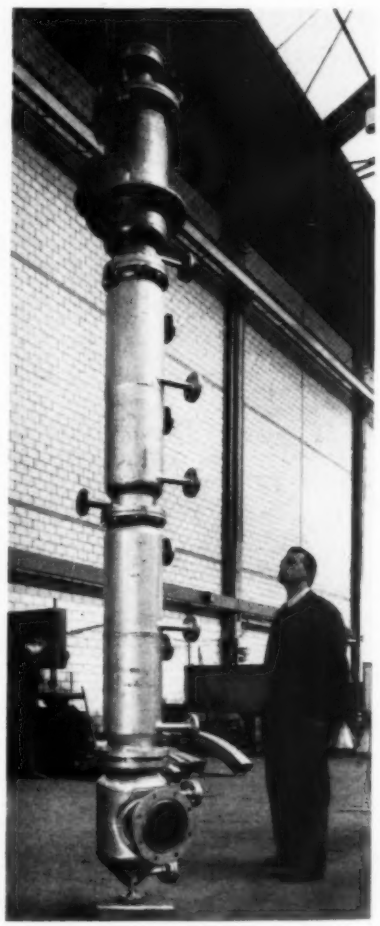
A new Transcope pressure transmitter with an indicating dial visible from 35 ft will soon be available. This pneumatic motion balance indicator can be servo-driven so as to provide ample power for process alarms and other auxiliary functions, with no loss of accuracy in the transmitted signal.

Transmitted signal is within ½ per cent of input signal. Indication is within ± 1 per cent of the actual value. Threshold sensitivity is 0.1 per cent of input span. Low friction drive mechanism permits accurate handling of low-energy input signals. The transmitter is designed for easy maintenance and has only three basic parts. It utilizes actuating elements which are common with Fulscope and other Taylor indicating, recording and controlling instruments. **Taylor Controls Ltd., 75 Hale End Road, Walthamstow, E.17.**

BCE 9840 for further information

New LUWA Fractionator

This fractionator has been developed for the efficient separation of mixtures of high-boiling, heat-sensitive substances. The unit separates multi-component mixtures at pressures of about 1 mm absolute pressure, with minimum pressure drop. Other features which are claimed are a large number of equivalent theoretical plates, high throughput and short hold-up.



DH-3 NOZLJECTOR

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"Valuable information Effendi. There is talk in the bazaars that Sharples are giving a complete chemical engineering service on any process connected with separation. A thousand thanks. You are my father and my mother, Effendi."

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P23

PLANT EQUIPMENT NEWS

The fractionator operates on the principle of alternate evaporation and condensation in a thin film and the unit may be operated continuously as a stripping or fractionating column, or in combination. A thin layer evaporator is used as reboiler. The equipment is supplied in the U.K. by **LUWA (U.K.) Ltd., Reliance House, 340 Clapham Road, London, S.W.9.**

BCE 9841 for further information

Membrane Permeation Cell

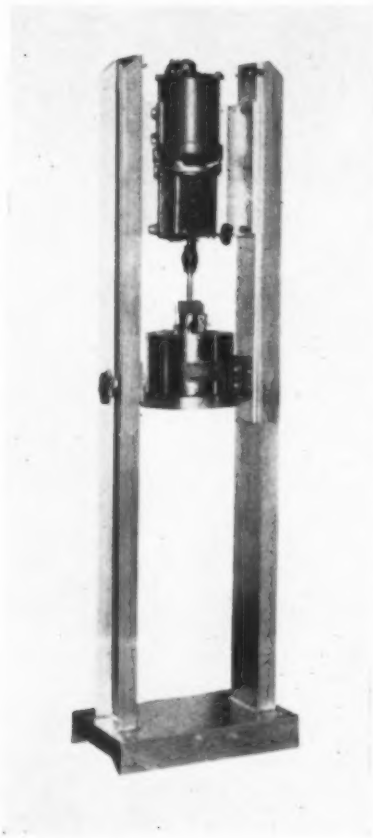
A new laboratory-scale membrane permeation cell for changing the composition and separating components of fluid mixtures is available from Ionics Inc. This new cell is claimed to make possible complete process research investigations of promising commercial application.

Metal parts in contact with fluids are of steel, but other materials can be supplied. Cell volume is 0.4 gal (1.55 litres), effective membrane area 0.165 sq ft (153 sq cm), standard rated working pressure 150 psig (11.6 kg/sq cm abs.) at 325°F (163°C). Higher pressure ratings can be supplied.

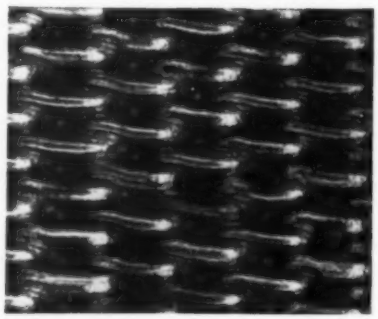
The cell is equipped with heating mantle, pressure gauge, thermometer well, and may be operated batchwise or continuously. An internal chamber provides a means of rapid cooling and a stirrer promotes uniform temperature and composition throughout the charge.

Optional accessories include a vacuum pump, condensers and glassware. Additional information from **Ionics Inc., 152 Sixth Street, Cambridge 42, Mass., U.S.A.**

BCE 9842 for further information



Wirecloth Filter Medium



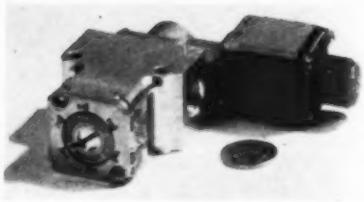
A new superfine filter medium is now being manufactured for the first time in this country. The woven stainless-steel wirecloth possesses a high degree of uniformity and retains particles of the order of 10 microns (0.00039 in.), as compared with 35.6 microns in 400/400 mesh, possible with the product previously pioneered in Britain by the same manufacturer.

The new material is twill woven with 0.0010 in. diameter wire, and a square inch of cloth comprises 325 warp wires running lengthwise, interwoven with 2300 tightly knocked-up weft wires, to provide 747,500 openings per square inch. Unlike square woven wirecloths, the openings in this Hollander specification are not apertures in the true sense of the word. Although absolutely uniform in critical dimension, they follow a sinuous path through the thickness of the cloth, do not permit transmission of light and permeation of liquids is only possible under pressure. The accuracy and regularity of limiting opening size is a major advantage of the material and its robust structure permits high operating pressures; being of stainless steel, it has excellent corrosion resistance and its application can cover a wide temperature range. The photograph shows the cloth known as 325/2300 Mesh Micro-Hollander Wirecloth, magnified 225 times. **The United Wire Works Ltd., Granton, Edinburgh, Scotland.**

BCE 9843 for further information

Pressure and Differential Pressure Switches

A small, lightweight device which opens or closes an electrical circuit when pressure, differential pressure or flow becomes too high or too low has been developed. These adjustable Deltadyne pressure switches provide reliable indication over the range from 15 to 200 psig. Within the pressure range of a given switch, actuation pressure may be altered by turning a direct reading dial.



These units can withstand system pressures or overpressures up to 5000 psi. When used as differential pressure switches, they exhibit actuation pressures which are completely independent of absolute working pressure. For example, a switch set to actuate at 30 psig will do so with 30 psig upstream and zero psig downstream, and will function equally well with 3000 psig upstream and 2970 psig downstream.

They can be used in all types of fluid lines including pneumatic systems and compressed gas lines, hydraulic systems, process streams, and other types of fluid system where an alarm device is required. Similar units are available with actuation pressures from 0.25 in. wg to 15 psi, and on special order from 200 psi to 5000 psi. **Pall Corpn., 30 Sea Cliff Avenue, Glen Cove, New York, U.S.A.**

BCE 9844 for further information

Expansion Joints for High-Pressure Ducting

A new type of restraint mechanism for accommodating thermal expansion in high-pressure and high-temperature piping systems has been developed. It will first be used with conventional bellows convolutions on the coolant gas ducting of Britain's new atomic power station at Trawsfynydd, North Wales (illustrated). Work is also in progress on design suitable for operating temperatures up to 600°C (1048°F).

The restraint, which works entirely on principles of elastic deformation, consists of a large number of high tensile bars, grouped in two frusto-conical assemblies round the periphery of the duct. In the axial direction it behaves as a stiff connection; angularly it can deform by transverse bending of the bars to a degree and at a rate which can both be accurately determined. And since the bars are uni-

BCE 9791 for further information

A metering pump

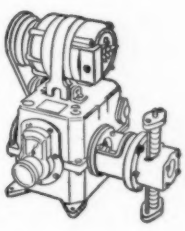
for Precision, Versatility, Durability

Metripump Type "G"

This Metripump is a precision, variable stroke metering pump of the positive displacement type, the length of stroke of which can be varied from zero to maximum either whilst the pump is in operation or at rest. A wide range of pump heads is available in different materials and with different designs of valve gear.

There is no substitute for experience in recommending metering pumps for specific problems.

Metering Pumps Limited

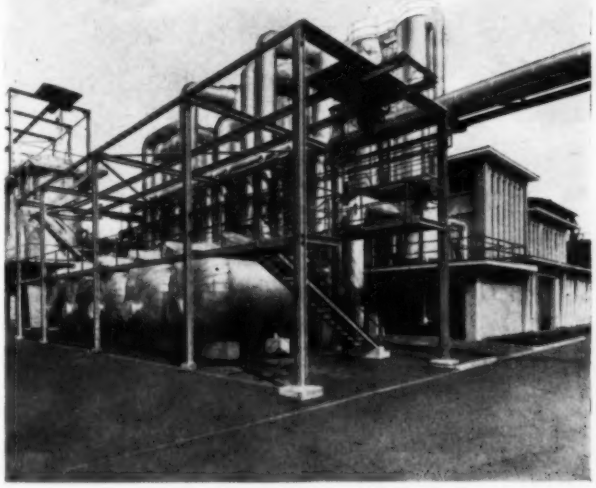


21 The Mall, Ealing, London, W.5.
Telephone: EALing 6264 (PBX)

BCE 9792 for further information

Carbon Monoxide Removal from Town Gas

R. & J. Dempster, Limited are pleased to announce that they can offer the Lurgi Process for Carbon Monoxide Removal from Town Gas.



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formly spaced round the periphery of the duct, the device is equally flexible in any direction.

A considerable range of deflections and restoring moments can be accommodated by varying the cone angle or the number and size of the tension bars. A centring device connecting the two ends of the duct on either side of the joint ensures that no torque loading can be applied to the bellows convolutions and equalizes the deflections of the two sets of bars under conditions of steeply changing bending moment along the duct. The shear force which accompanies such a bending movement is taken entirely by the device, none being transmitted to the bellows. The tension-bars, which are made of the highest quality steel and rigorously tested, must be able to withstand total gas pressure loads of well over 1,000,000 lb (453,592 kg). **Richardson Westgarth & Co. Ltd., P.O. Box 2, Wallsend-upon-Tyne, Northumberland.**

BCE 9845 for further information

New Chemical Pump

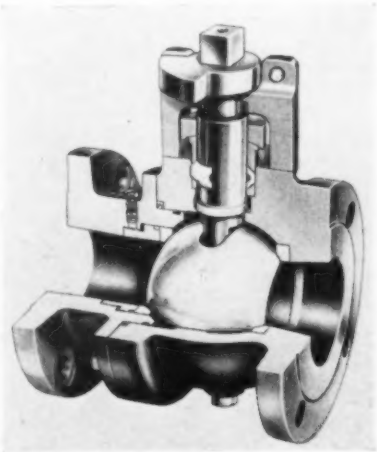
Diafilter Engineering Co. Ltd. will shortly be marketing a positive-displacement diaphragm pump capable of delivering from 2.0 to 20 gal per 24 hr by single control adjustment. It can apply against pressures up to 100 psi without the need for a balancing water supply. All components of the head of the pump are manufactured from corrosion-resisting materials. The head is of clear plastic so that the pumping action can be observed.

Glassfibre-reinforced polyester is used for the body, housing a fan-cooled motor of only 60 watts consumption and available for 200/250 or 110 volts. The instrument is 10 in. long and 8 in. high and weighs only 8 lb.

The new pump, to be known as the Diachlor 361, is complete with all accessories when sold. These are suction and delivery tubing, point of application fitting, solution strainer assembly, cable and three-pin 5-amp plug. **Diafilter Engineering Co. Ltd., 902 Purley Way, Purley, Surrey, England.**

BCE 9846 for further information

Ball Valves



These consist of a spherical ball "floating" in resilient seats which provide a positive shut-off in either direction. The ball has a cylindrical hole which provides a round straight-through port when the valve is open.

No sealing lubrication is required and the seals are protected from flow abrasives allowing the valves to handle slurries.

Ball valves are quarter-turn from full-open to full-closed and therefore quick acting. They are of simple design with few moving parts, and there is little friction and wear.

Valves manufactured cover both flanged and screwed-end types to B.S.S., A.S.A. and A.P.I. standards. They are available in a variety of body materials (cast iron, cast steel, bronze, aluminium, stainless steels, etc.) combined with several different types of sealing, such as Teflon, Viton, Buna-N, etc., to cover a wide range of services. **Weir-Pacific Valves Ltd., 95 Coltness Street, Glasgow, E.3.**

BCE 9847 for further information

Digital Read-out Differential Refractometer

This new instrument uses a null meter and digital read-out to measure index of refraction changes as small as 0.0000005 unit. The instrument can be set such that 1000 units on the digital read-out is equivalent to a R.I. range as narrow as 0.0005 unit or as wide as 0.2 unit.

Instruments can be supplied for a fixed factory calibrated range and with accessories to obtain any range between 0.0005 and 0.2 unit.

The digital read-out is graduated from 000 to 1000 and can be read to 1 digit. One of the two models (Model 107B) is for quality control of batch or continuous processes. It is factory calibrated and portable. A recording version is also available. **Scientific Furnishings Ltd., Poynton, Cheshire.**

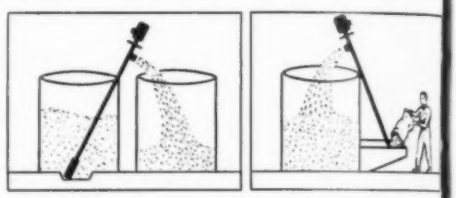
BCE 9848 for further information

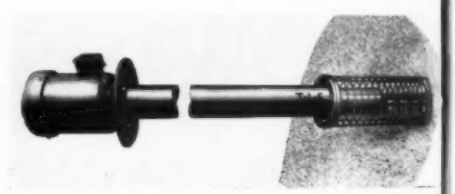
Teles Super Elevator

An inexpensive, direct-driven elevator with a single-piece auger now available has an exceptional performance compared with other machines of the same type. It will handle up to 10 tons/hr of granular materials and the unit, which has a 3-in.-dia., 11-ft shaft, may be extended if required. Extensions are available in 5- and 10-ft lengths and any number may be bolted together.

If the conveyor is likely to be used for a number of products, a reverse starter can be provided which enables the auger tube to be completely cleared of material before another different material is handled.

Another useful feature is the special





intake control which prevents granular material from jamming the machine, and which allows the flow rate to be controlled. **Teles Smith Ltd., 8 South Wharf, Paddington, London, W.2.**

BCE 9849 for further information

Light-duty Gear Unit

A new Light-duty gear unit has been produced for use with certain wrench-operated Audco valves. The unit has been so designed that it may easily be fitted to existing valves already in service, or it can be supplied assembled on a new valve.

This unit has a cast-iron quadrant which is clamped to the plug stem of the valve. No lubrication is needed on the gear, a better performance being obtained when dry.

It offers an inexpensive mechanical means of operating certain larger wrench-operated valves with a minimum of effort. **Audco Ltd., Newport, Shropshire, England.**

BCE 9850 for further information

BCE 9793 for further information

A revolutionary development in SURFACE HEATING

Hotfoil Flat-Element Heating Tapes introduce a new conception of heat transfer efficiency. Loadings more than twice those obtained with round-wire conductors are achieved through the use of metal foil elements insulated with high temperature plastic material and woven glass.

Hotfoil Tapes provide a most effective method of compensating for heat losses in pipe lines and raising temperatures in tanks or vessels containing almost any fluid. They can also be used for protecting steam and water pipes against frost.

Robust yet flexible, Hotfoil Tapes have an infinite variety of other applications in industry.

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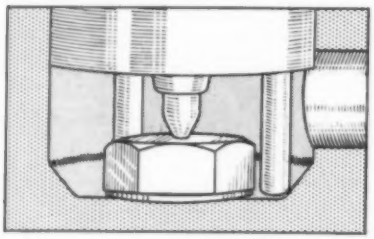
PLANT EQUIPMENT NEWS

Thermostatic Expansion Valves



A special feature of these valves is a new natural stone corrosion-free needle and double-contact bulb. The special adsorption charge leaves the thermostatic element unaffected by temperature variation of the valve body. The regulating ability of the valve cannot be disturbed, although it may be fitted any way up and in any position. The all-metallic type TV has a superheat range of between 2°C and 24°C at an evaporating temperature of −10°C. **Danfoss Automatic Controls and Equipment, Nordborg, Denmark.**

BCE 9851 for further information

Limpet Pumps

Versatile, self-priming units which can be used for a very wide range of duties. Capacities up to 545 igpm (2470 litres/min) and heads up to 135 ft (41 metres). These pumps have an open impeller and are particularly useful for pumping dirty fluids found in bilge, ballast, irrigation and draining. In most marine applications, and where considerable movement is encountered, an extended suction cover is fitted. Foot or flange mounted pumps are available and can be fitted with differing types of seals or glands. **Hamworthy Engineering Ltd., Poole, Dorset.**

BCE 9852 for further information

Industrial Electronic Weighing

A new range of electronic weighing equipment is being introduced into Great Britain. A full engineering service is provided to cover design and installation of complete systems including instrumentation and associated steel structures.

The equipment will indicate and/or record weights, and equipment can also be provided to drive teletype and punch card accounting equipment.

The system can be integrated into process control schemes. It is particularly adaptable for use in batch weighing, tank and hopper weighing, crane weighing and weigh-bridge systems. It is robust and can operate under severe conditions. **Rotax Ltd. (Process Control Group), Chase Road, London, N.W.10.**

BCE 9853 for further information

Hot Air Plastic Welding

A comprehensive range of welding torches and auxiliary tools is now available for welding thermoplastic materials, including polypropylene.

The basic instrument is the electric hot air plastic welding pistol giving a heat range from 250-600°C.

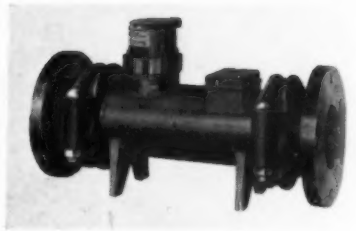
Another instrument is the LIF hot air forming tool (illustrated) which is self-contained with motor, blower and 2000-watt heating element. An air output of 19 cfm at 400°C is obtained and enables bending, pre-heating, etc., to be easily carried out. **Welwyn Tool Co. Ltd., Stonehills House, Welwyn Garden City, Herts.**

BCE 9854 for further information

Versatile Water Meters

Known as the type 33, a range of new full-flow water meters designed for use in any pipeline having a high flow rate and requiring minimum heat loss conditions, is available.

Four sizes of the new meter can handle flows of from 800 to 200,000 Imp. gph. They have simple two-bolt Victaulic joints for easy installation in any pipeline. Operation is based on a helical rotor mounted in plastic bearings on the meter body axis. Maximum error is not more than ±2 per cent. **Parkinson Cowan Measurement Ltd., P.O. Box 2, Dobcross, Nr. Oldham, Lancs.**



BCE 9855 for further information

Goodyear B.12 Pump

On show for the first time at the *Engineering, Marine, Welding and Nuclear Energy Exhibition, 1961*, was an entirely new pump developed from the successful A.12 model. It can be stripped without disturbing the pipe joints and can be fitted with the Goodyear automatic by-pass relief valve which has a hand-unloading valve incorporated. Available in bronze or stainless steel, the latter will be of great interest to chemical engineers. Speed range is from 500 to 3000 rpm. **Goodyear Pumps Ltd., 44 Brook Street, London, W.1.**

BCE 9856 for further information

Molecular Sieve Driers

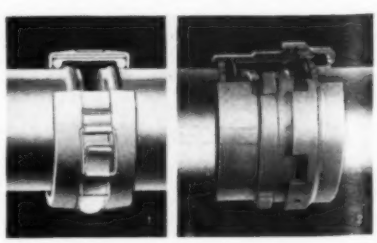
This range of free-flow driers, produced by Dean & Wood, was exhibited at the recent *International Refrigeration and Air Conditioning Exhibition*. They meet the need for an efficient drier, using molecular sieves as the drying agent for moisture removal and embodying a strainer which will trap and hold large quantities of foreign particles. Through co-adsorption, molecular sieves will remove inorganic and organic acids present in the system. By removing moisture and acids simultaneously, they eliminate acid-corrosion problems. **Dean & Wood (London) Ltd., 17a-19 Artillery Row, Victoria Street, London, S.W.1.**

BCE 9857 for further information

Flexible Couplings

Three new types of flex coupling, originally designed for aeronautical use, are now marketed under licence in the U.K. and Commonwealth. These are intended normally for fuel, hydraulic, pneumatic and lubrication systems, but can be adapted for corrosive fluids.

Main feature is that they permit axial and angular deflection of the pipes and a misalignment of up to $\frac{1}{16}$ in. is acceptable when fitting. Tubes are flared at the

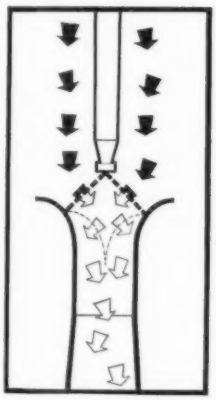


BCE 9794 for further information

"DOUBLE SCRUBBING"

IS THE ANSWER

to really efficient gas cleaning



In the Ambuco venturi scrubber, made under licence from Waagner-Biro, the gases are compelled to pass through *two* complete cones of water. This exclusive feature guarantees up to 99·6% collection efficiency of most entrained solids in the range 5 to 0·01 microns.

Send for fully descriptive bulletin.

The first cone of water removes the coarser solid particles; the second cone ensures that even the very finest are collected. The position of the water injection nozzle can be varied to obtain a droplet size to suit any set of conditions.

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BCE 9795 for further information

What's going on here?

A North PVC apron, for safety's sake

North PVC aprons are absolutely made for jobs that are chemical, oily, greasy, rough or tough. North PVC aprons last longer, cost less than you'd expect. North PVC aprons are—but why not write for a leaflet, a price list, and see for yourself why industry after industry specifies these aprons, and nothing but. Which reminds us: North PVC protective garments, headgear, gloves, and other etceteras—we have plenty of booklets about them, too. Let us send you the lot.

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PLANT EQUIPMENT NEWS

ends to prevent pulling out through the seal.

The three new types comprise: (1) a lightweight coupling with a twin beaded seal; (2) a screw type with "C" ring seals, mainly for aeronautical applications; and (3) a heavy-duty type for industrial applications. **Aero Controls Ltd., Industrial Estate, Weedon Road, Northampton, England.**

BCE 9858 for further information

Packaged Liquid Chillers

The Chrysler liquid chiller unit incorporates dry expansion-type liquid cooler, efficient shell and tube condensers and accessible radial design compressor. There are dual circuits in the cooler affording complete control and allowing the cooler and compressor to operate at reduced capacity. The available units will cool water from 52-42°F in quantities ranging from 38-248 gpm. **Chrysler Corporation, Airtemp Division, Dayton, Ohio, U.S.A.**

BCE 9859 for further information

Electric Melting Kettle

A standardized heater for heating high-viscosity materials to a pouring state is now made in quantity.

It was specially adapted to melt carbon wax and is complete with thermostatic control. The heated hose and valve are controlled by the same thermostat that controls the contents, thereby allowing the contents to drain completely and clearly.

The kettle has a total capacity of 10 gallons and is heated by the standard Stabilag multi-tiered heating zone principle with a total power of 5½ kW. The unit is very manœuvrable.

The price of this 10-gallon unit complete is £238 with a delivery of one month. Specials can be made to order and a standard 20-gallon unit will be available at £275. **The Stabilag Co. Ltd., Mark Road, Hemel Hempstead, Herts.**

BCE 9860 for further information

Murphy Transweigh System

The Transweigh patented system* for electrically weighing and controlling belt-conveyed solids is now available in the U.K. and Western Europe through Murphy Radio. It is claimed that bulk materials in transit can be measured and controlled with the ease and accuracy of fluid flows. Practically any type of automatic control can be obtained: from the regulating of feed rates to continuous blending systems. Transweigh equipment has been installed in mining, steel production, power generation, paper manufacturing, cement production and chemical processing. **Murphy Radio Ltd., Electronics Division, Welwyn Garden City, Herts.**

* British Patent No. 738,353.

BCE 9861 for further information

New Publications

Bibliography on Water Conservation

The first comprehensive bibliography of references to literature on the subject of water conservation lists 682 references—the work of 564 authors and authorities all over the world. It is No. 7 in a series of Technical Publications, and will be made available free of charge to authorities and research workers concerned with this work.—**Price's (Bromborough) Ltd., Bromborough Pool, Bebington, Wirral, Cheshire.**

BCE 9862 for further information

A four-page leaflet published by Imperial Aluminium Co. Ltd. describes the properties of Impalco roll-welded transfer sheets. These sheets are made by pressure welding selected areas of two aluminium sheets during cold rolling and are claimed to offer a highly efficient heat transfer medium. There are no joints to impede heat transfer and the tube pattern can be designed to meet almost any requirement.

Applications include refrigerator evaporators, cooling electronic equipment, milk and beer coolers, panel heaters and many others.—**Imperial Aluminium Co. Ltd., P.O. Box 216, Witton, Birmingham.**

BCE 9863 for further information

A range of data sheets, describing over 225 items of 20 basic types of standard woven wire mesh element-in-line filters, has been published by Aircraft Porous Media Inc., a subsidiary of Pall Corporation. Each of the series of 20 data sheets shows a cross-sectional view of the filter element and housing. Working pressure, temperature ranges, port sizes and other factors are listed for each type shown. Optional variations are shown for each basic type, allowing many combinations for varieties of applications.

Standard units range in tube or pipe sizes from ¼ in. to 12 in. A variety of fluids, ranging from cold gases to hot viscous polymers in the temperature range of −320°F to +1000°F., may be handled. —**Pall Corporation, Glen Cove, New York, U.S.A.**

BCE 9864 for further information

Hidumium 400, a new thermal neutron shielding material, is the subject of a booklet published by High Duty Alloys Ltd. The product consists of boron carbide of fine mesh size which is evenly dispersed with a fully wrought aluminium matrix and clad on both sides with aluminium of high purity. The principal advantage of the new material is its greatly improved formability.

Full details of the typical mechanical properties of Hidumium 400 are given, in addition to welding, shearing, tapping, drilling and punching properties.—**High Duty Alloys Ltd., Slough, Bucks.**

BCE 9865 for further information

Shaker Furnaces. Shaker-hearth furnaces are the subject of a new publication from Efco Furnaces Ltd. The furnace is designed for the continuous heat treatment of small steel parts and can be incorporated with continuous production lines. Bright hardening and shallow case hardening of such items as pins, needles, watch components and ball and roller bearings can be carried out.

The hearth is in the form of a hot plate and remains inside the furnace so that thermal efficiency is high.

Several quench arrangements are available and hand-operated or conveyorized discharging gear is available. The various combinations of oil and salt quenching arrangements are shown clearly by means of diagrams.

A junior model to deal with small quantities of about 15 lb per hour is available in addition to the standard model. **Efco Furnaces Ltd., Queens Road, Weybridge, Surrey.**

BCE 9866 for further information

Sondes Place Research Institute. The objects and scope of the Sondes Place Research Institute are described in a new publication. The Institute carries out sponsored research and development work in chemistry, chemical engineering and mechanical engineering. It started operations in 1936 as a consulting chemical and chemical engineering practice and was incorporated as a limited company in 1947.

General policy of the Institute is to avoid excessive specialization in particular industries and to apply basic scientific knowledge to solving practical problems in a wide field.

The bulk of the work of the Institute comes under the following headings: information, project assessment, laboratory research, analysis and testing pilot plant development, plant design, mechanical prototyping. **Sondes Place Research Institute, Dorking, Surrey.**

BCE 9867 for further information

"The Yarsley Organization" is the title of a booklet published by Yarsley Research Laboratories Ltd. It describes the origins and scope of the company with the help of a number of excellent illustrations.

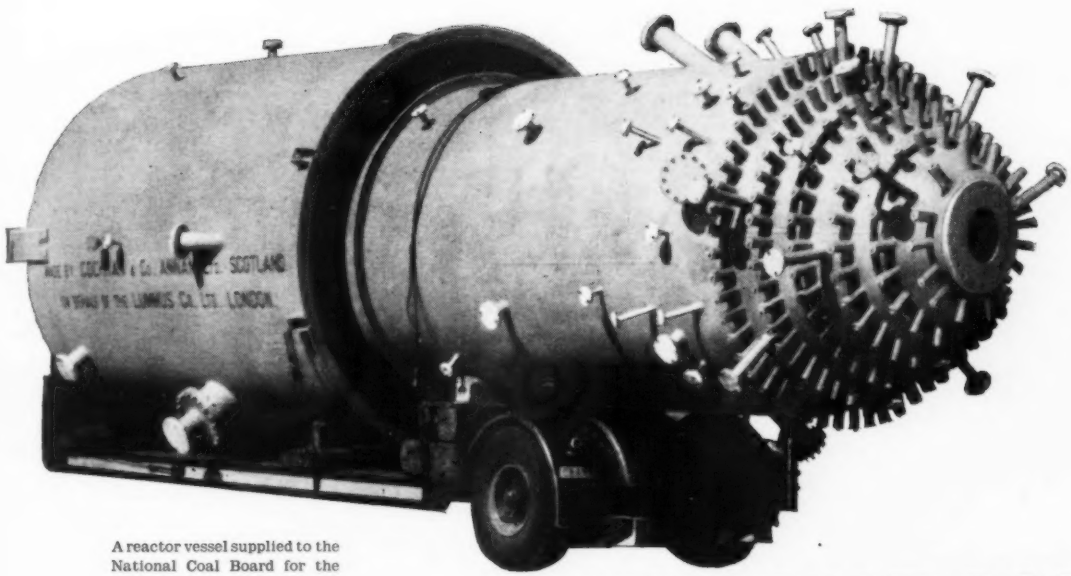
The company was formed in 1931 to give a consultative service to the plastics industry and its chief concern is now still with plastics, rubbers, adhesives and surface coatings. A confidential consultative service is given to clients, whose names are not disclosed, on a wide range of problems, including practical investigations on processing and product quality, fundamental and applied research, patent and economic surveys, and testing and analysis of a wide range of industrial products and consumer goods.

Care is taken by the company to avoid entering into any commitment with more than one client on a similar problem or theme. **Yarsley Research Laboratories Ltd., Clayton Road, Chessington, Surrey.**

BCE 9868 for further information

BCE 9796 for further information

PRESSURE VESSELS



A reactor vessel supplied to the National Coal Board for the production of smokeless fuel.

BY COCHRAN

For years, this Company has built Class I fusion welded Pressure Vessels; Cochran & Co., Annan, Ltd., were in fact one of the first half-dozen manufacturers in this country to be approved by Lloyds for this class of work. Cochran's Pressure Vessels are built to all codes of construction in a wide range of sizes and materials, in plate thicknesses up to 2½", with a maximum one-piece weight of 110 tons. Into these Pressure Vessels go the skill and experience which have long been associated with the Boilers—vertical and horizontal, coal and oil-fired, packaged and conventional—which have made famous the name of COCHRAN.

An autoclave for the steam curing of reinforced concrete sections.

COCHRAN & CO., ANNAN, LIMITED • ANNAN, DUMFRIESSHIRE, SCOTLAND • PHONE : ANNAN 111 • OFFICES IN LONDON ABBEY 4441 & GLASGOW CENTRAL 4929

AP15

World of Chemical Engineering

New Processes

Fatty Alcohol from Oils and Fats

The Hungarian Institute for High-Pressure Research has developed a new process for obtaining fatty alcohol from various animal or vegetable oils and fats. By means of high-pressure hydrogenation (300 atm) and in the presence of a special catalyst, the fatty acids from the oils and fats are transformed into fatty alcohol. The alcohols are then separated by distillation from the non-transformed raw material. They are converted, by a sulphating operation, to fatty alcohol sulphates, and these highly effective washing materials, mixed with other substances, are marketed as bleaching powder.

Production of High Purity Water

Bogue Electric Manufacturing Co. have developed an industrial process which produces water containing less than 10^{-9} parts of foreign matter. This water is used in the manufacture of electronic equipment and components. The process converts soluble matter, by ion exchange, to compounds which are easily removed. The plant is continuous and produces 100 gpm of water whose purity is measured electrically in ohms. Calculations indicate that absolutely pure water would have a resistance of 24×10^6 ohms per cm^3.

Phthalic Anhydride from New Source

HUGH HARPER, of the Scientific Design Co. Inc., described, in a paper presented at Manchester University, how it was possible to obtain phthalic anhydride yields from ortho-xylene by a new SD process. The yields of anhydride were comparable to those obtained from naphthalene in the U.S.A.—85 to 90 lb per 100 lb of ortho-xylene. The appreciable amount of maleic anhydride produced by the ortho-xylene oxidation can be recovered and converted inexpensively to fumaric acid. Phthalic anhydride can be produced from ortho-xylene in the same plant at the same capacity, from the same reaction system and catalyst that is used for naphthalene.

Maleic anhydride is to be manufactured by a process based on the air oxidation of benzene. Scientific Design Co. are building the plant at Colleferro, Italy, for Bombrini Parodi-Delfino S.p.A.

A British firm, Grange Chemicals Ltd., has plans to produce phthalic anhydride from ortho-xylene instead of from naphthalene. A new plant to manufacture 15,000 tpa is to be built at BP's Kent refinery.

New Plants in Brief

The U.S.S.R. is to build a plant for the production of ammonia-based liquid fertilizers. It will be part of the most modern chemical combine in the Soviet Union. Other important expansion projects include those at the Gorlovka works, where nitrogen capacity is being trebled; the Chirchik electrochemical works, where nitrogenous fertilizer production is due to start shortly on the basis of Bukhara natural gas; and new nitrogen plants at Grodno, Nevinnomyssk and Rustavi.

New ammonia and nitric acid plants have been announced at home and in Spain. Esso and Fisons are to build a £4 million ammonia plant at Milford Haven, adjacent to the new Esso refinery. It will produce 150,000 tpa of ammonia. Cia Abonos de Sevilla, in association with Soc. Ansaldo of Genoa, are to construct a 225 tpd nitric acid plant to be on stream in 1963.

A sulphuric acid plant and a single superphosphate plant are to be supplied by Simon-Carves Ltd. as part of a new fertilizer factory, which is to be built for the Uganda Development Corporation Ltd.

In the metallurgical field important developments have been carried out by The International Nickel Co. of Canada Ltd., who have increased their annual nickel producing capacity by more than 75 million lb, bringing it to more than 385 million lb with the opening of their Thompson project. The British Steel Industry at Scunthorpe are to have a new oxygen plant built by The British Oxygen Co. Ltd. It will increase capacity at their Warren-lane works to 600 tpd.

Acetylene is to be produced by E. I. de Pont de Nemours & Co. for its own use at a new plant to be built at Montague, Mich. A modification of the German Hüls electric arc process is to be used. Only 3½ kWh/lb of electricity will be required, instead of about 5 kWh/lb needed in other arc processes.

Midland Silicones Ltd. are to spend £1 million on plant expansion at Barry, South Wales. This will include new plant to manufacture emulsions for use as release agents and for the treatment of textiles and paper. A methylchloride plant and a new direct process unit for the production of chlorosilanes have just started production. The illustration shows intermediate storage and handling equipment on the new silicone fluids plant.

Among the large projects approved by I.C.I. during the year 1960 were plants for the manufacture of nylon, chlorine, polypropylene filament yarn and various petrochemical products and plastics. The company also intends to manufacture caprolactam, the monomer for nylon "6" polymer. A plant capable of distilling about one million tons of crude oil annually is to be built on a site on the north bank of the River Tees. It will cost several million pounds.

Other new petrocarbon plants are being developed by British Hydrocarbon Chemicals Ltd., who have announced that the initial operations at the Baglan Bay works will be based on a steam cracker, producing the olefins, ethylene, propylene and butadiene from the resultant mixture of cracked gases. Also, Chemische Werke Hüls AG announce that work will soon begin on the construction of a plant for the production of synthetic rubbers based on butadiene and isoprene.

The Haifa Refineries (Israel) are to invest I£4 million to reactivate one of their three cracking and distilling units. Four petro-chemical plants, costing $16 million, are to be built near the refineries. Carbon black, ethylene, polyethylene and DDP will be produced.

Organic chemicals production capacity has been increased. The only producer of trichlorethylene perchlorethylene in Sweden, The Uddeholm Co., can now supply all the demands of the Swedish market from the new automated plant at Skoghall. The manufacture of organic chemicals by The Houdry Process Corporation is a new field of activity. One of their products is triethylenediamine (known as Dabco), which is used as a catalyst in the manufacture of polyurethane plastics.

The fabrication of large constructions in rigid P.V.C. and all Orglas resin/glass laminates will be undertaken at a new unit to be erected by Prodorite Ltd.

Company News

Thomas Broadbent & Sons Ltd., of Huddersfield, announce that they have appointed Messrs. Thomas & Carroll Pty. Ltd., Chemical Engineers, of Melbourne as representatives in Australia for the complete range of chemical centrifuges now in production.

Luwa (U.K.) Ltd., a British subsidiary of the well-known Swiss firm, have commenced business at Reliance House, 340 Clapham Road London, S.W.9 (MAC 7776), as suppliers of plant based on the Luwa thin film evaporating machine and the rotary high-vacuum fractionator.

Dewrance & Co. Ltd. announce the formation of a metals division. It will be responsible for the production and sales of high quality nonferrous casting, nickel alloy castings, stainless steel castings and high-temperature brazing alloys.

Fisons Fertilizers Ltd. have formed a new manufacturing company in association with Union Chimique Belge of Brussels. To be known as Fison U.C.B. S.A., the company will produce high-analysis granular compound fertilizers at a manufacturing plant to be built near Ostend.

Nuclear Developments Ltd. is the name of a new company formed jointly by I.C.I. Ltd. (Metals Division), Rolls-Royce Ltd. and the Rio Tinto Co. Ltd. It will operate in the field of civil nuclear engineering in collaboration with the atomic power consortia.

Contracts

Imperial Chemical Industries of Australia and New Zealand Ltd. have ordered a small but powerful electronic digital computer from Ferranti Ltd. for use in their Central Research Laboratory in Melbourne.

Uniao Fabril do Azoto, Portugal, are to erect a new fertiliser project near Lisbon. It will consist of an ammonia plant (180 tpd), a urea unit (40,500 tpa), a nitric acid plant, an ammonium nitrate plant and a nitro-limestone plant. The Kellogg Co. of New York have been awarded contracts covering the ammonia and urea plants.

W. H. Allen, Sons & Co. Ltd., Bedford, have received an order from the Lummus Co., covering the supply of a diesel-alternator set for a synthetic rubber plant being built in India.

CIBA will make available to Humphries & Glasgow and Atul their manufacturing processes and know-how for the Atul Products Ltd., India, expansion programme in the dyestuffs field.

R. & J. Dempster Ltd., of Manchester, have received an order for a Stretford Sulphur Extraction Process Plant from the Eastern Gas Board.

Millspaugh - Wimpey Ltd., London, have been awarded a contract worth more than £3 milion for the establishment of a complete paper mill in Yugoslavia. It includes the design and supply of all plant and equipment as well as technical assistance in the erection of the mill. It will provide 120 tpd and will be located at Plaski in Croatia.

Simon Carves and I.C.I. have received contracts worth £7 million for the design and partial erection of four polythene plants in East Germany, Czechoslovakia, Poland and Rumania.

A gas-oil blending plant is to be supplied to J. C. Oxley's Dyes & Chemicals Ltd. by the Blaw Knox Co. Ltd.

Process Plant Contractors (Campbell) Ltd., Manchester, have been awarded a £½ million contract for the design, supply and construction of an acid recovery and storage plant for British Sidac Ltd.

Personalities

Mr. W. F. Gerrard, F.R.I.C., has resigned from the Liverpool Borax Co. Ltd., and has been appointed Managing Director of the Atlantic Water Treatment Co. Ltd., Liverpool.

Mr. B. C. Morton, M.I.Mech.E., M.Inst.Gas.E., has been appointed Deputy Chairman of Robert Dempster & Sons Ltd. Mr. Charles Dempster and Sir Robert Perkins have retired from the Board.

Gustave F. Wunder has been appointed General Manager of Procon (G.B.) Ltd., refinery and chemical plant construction company, a subsidiary of Procon Inc. (U.S.A.).

Mr. T. Watts, B.Sc. A.M.C.T., A.R.I.C., A.M.I.Chem.E., has been appointed Senior Chemical Engineer in the design department of The Technical Division of Process Plant Contractors (Campbell) Ltd. Mr. D. Emmett, B.Sc., has joined the same Division as a Senior Design Chemical Engineer. He was previously with British Titan Products Ltd.

Mr. Theodore Q. Eliot, a chemical engineering graduate from MIT, and a member of the American Institute of Chemical Engineers and American Chemical Society, has joined Scientific Design Co. Inc. as Sales Liaison Engineer in the Project Evaluation Department.

British Trade Fair in Sweden

The largest all-British Fair ever staged abroad under the auspices of the Federation of British Industries is to be held in Stockholm from May 18 to June 2, 1962. It will form part of a major drive to increase British exports to E.F.T.A. countries. There will be more than 100,000 sq ft of covered space and 175,000 sq ft of open-air space available. Basic rates for interior sites are 28s 6d per sq ft; for exterior sites they are 12s 6d per sq ft. Applications to British Overseas Fairs Ltd.

Problems Solved

Pumping Phosphate Sludges

Pumping of corrosive and abrasive solutions is an operation that frequently presents difficulties. An example where non-metallic materials of construction have been used is provided by a recent installation reported by Union Carbide. The pump had to handle abrasive phosphate sludge with concentrations of phosphate in the ranges from ½ to 4 per cent by volume with pH ranging from 1.5 to 5. Another pump was required for handling acid, alkaline and solvent emulsions at temperatures up to 93°C. Impervious graphite pumps proved to be the answer to both problems, and so successful were they that after two and a half years' service the head pressure (205 psi) had shown absolutely no variation from the time of installation.

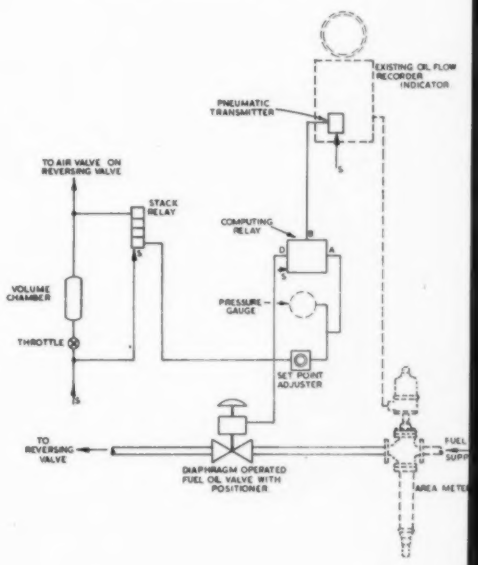
Computing Relay in Fuel Control

The following are the main features of a control system applied successfully to a Manchester steelworks. This system was devised to enable the furnace to be fired alternately from one end and then the other while the fuel oil flow to each furnace is determined by the operator who adjusts the set point of the computing relay; the relay controls the action of a valve controlling fuel supply. The system used for the latter purpose is shown in the accompanying diagram.

When the reversing valve is in the normally open position, the air valve, which is operated by the action of the reversing valve, is shut. This allows the system to operate under the normal control of the fuel oil flow loop.

When the reversing valve is shut, the air valve is open, which then bleeds air to atmosphere. The pressure in chamber "A" of the stacked relay goes to zero, thereby shutting off the air supply to the control system and closing the fuel oil control valve.

Continued on next page



When the next cycle begins, the reversing valve opens and the air valve closes. This then allows air pressure to build up at a rate determined by the throttle adjustment. As this pressure increases, the pressure in chamber "A" of the stacked relay is increased, thereby transmitting a steadily increasing set point signal to the computing relay, which slowly opens the diaphragm control valve and avoids furnace "puffs" which can occur when firing is commenced too suddenly after a reversal.

Reduced SO_2 Emission from Contract Plants

Pollution regulations in some areas of the United States require that at sulphuric acid plants the stack effluents analyse less than 0.20 per cent SO_2. At one plant belonging to the Stauffer Corporation it was found that the stack effluents analysed 0.40 per cent SO_2. This meant that some action had to be taken to bring the content of SO_2 below the legal limit. Engineering studies proved conclusively the necessity for lesser space velocities within the catalyst beds, and for favourably shifting the SO_2-SO_3 equilibrium ratio by air addition, if 0.20 per cent sulphur dioxide exits were to be realized. Accordingly, as much catalyst as practical for pressure drop limitations was added, and sulphur dioxide concentrations were decreased from 9.5 per cent to 8 per cent. As predicted, the sulphur dioxide portion of the tail gas was halved.

Subsequently, through increased demand for sulphuric acid it became necessary to extend the plant and, in order to ensure that the SO_2 emission did not exceed the specified limit, a fourth catalyst bed in the converters became necessary. The reduced SO_2 concentration required an increase in the size of the converter exchanger and flues by some 20 per cent in order to maintain output rates and avoid high pressure drops within the units. The increased cost of the installation, caused by the legal requirements, has been partly met by the increased recovery of sulphur.

Another problem encountered by the same company was the emission by natural breathing of strong sulphur dioxide from spent alkylation acid storage tanks. This was overcome by purging the tanks with a slip stream of stack gas. The danger of flashback was overcome by installing a flame arrester, using a low-oxygen purge gas, and by installing an electrically operated anti-flashback system which incorporated check valves.

These problems and their solutions emerged from a paper entitled "Air Pollution as it concerns the Sulphuric Acid Industry in the Los Angeles Area", given by RICHARD V. WALLACE and LESTER H. WITTENBERG, of the Stauffer Chemical Co., at a recent A.I.Ch.E. meeting.

Control of Process Water Quality

In order to ensure that the supply of water from mains to be used in a food processing plant was absolutely free of sediment and suspended matter, a special system had to be devised which would detect water with undesirable qualities and allow it to be diverted from the process feed tank. Concentrations of the order of a few parts per million had to be detected and reliability was an essential quality of the instrument to be used. These conditions were fulfilled by a photocell system which was provided with high sensitivity by the incorporation of variable potentiometers in the control unit; this was installed in the water line supplying the tank. So that immediate action can be taken when excess turbidity occurs, a series of three remote warning panels are located in the factory with alarm bells and indicator lights. When an alarm sounds an operator depresses a muting switch, which stops the audible warning but which maintains the visual red light warnings. The latter persist until normal water conditions are restored. In the future it is expected that the whole plant will be made fully automatic by the use of motorized and magnet valves. This installation was the work of Radiovisor Parent Ltd.

Errata (May issue)

Adsorption Processes,
by J. W. CARTER

In Fig. 2 of this review, acknowledgment should be made to *Chemical Engineering News*, and Fig. 4 of the review should have been Fig. 7 of Reference 25, and not Fig. 6.

Fluidization,
by J. S. M. BOTTERILL

Two points in this review require clarification. On page 328 it is suggested that the insertion of baffles narrows the residence time spectrum "to that of a perfectly mixed unit". The latter phrase should be "to that of a series of perfectly mixed units".

Also the effect of injecting water (second paragraph, left - hand column, page 329) was to cause "aggregation" rather than "segregation".

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Temperature Dependence of the Binary Diffusion Coefficient of Gases
D. S. SCOTT, K. E. COX
Can. J. Chem. Eng., 1960, **38**, 6, 201.

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D. D. KALAFATI
Teploenergetika, 1960, **1**, 72.

Applied Hydrocarbon Thermodynamics (Part 25)
W. C. EDMISTER
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P. R. STEYERMARK *et al.*
Ind. Eng. Chem., 1961, **53** (3), 212-14.

Effects of Alternating Currents in Causing Corrosion
F. E. KULMAN
Corrosion, 1961, **17** (3), 34-5.

Determining True Residence Time in Flow System Reactions
M. R. HOARE
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Calculating Activity Coefficients
M. G. MYLES, R. E. WINGARD
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Combustion Nuclei—An Index for Classifying Gaseous Hydrocarbon Fuels
J. E. QUON *et al.*
Ind. Eng. Chem., 1961, **53** (3), 235-8.

Kinetics and Mechanism of Zinc Dissolution in Acid
R. S. KAPNER, H. E. HOELSCHER
Ind. Eng. Chem., 1961, **53** (3), 239-44.

Size Reduction—A Review
L. T. WORK
Ind. Eng. Chem., 1961, **53** (3), 245-7.

High-temperature Distillation—A Review
T. J. WALSH
Ind. Eng. Chem., 1961, **53** (3), 248-50.

Mass Transfer on Bubbler Grid and Tube Plates of the Drop-through (Turbogrid) Type
I. N. KUZ'MINYKH, A. I. RODIONOV
J. Appl. Chem., USSR, 1959, **32** (7), 1523-8.

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I. SAMOIL
Rev. de Chim (Rumania), 1961, **12** (2), 65.

Continuous and Automatic Analysis of Streams Process by Gas Chromatography
H. J. MAIER, H. N. CLAUDY
Ann. N.Y. Acad. Sci., 1960, **87** (2), 864-74.

The Use of H_2SO_4 for Regeneration of Cation-exchanger Beds in Complete Demineralization of Water
U. P. MELESHKO
J. Appl. Chem., USSR, 1959, **32** (6), 1264-71.

The Design of Automatic Trace Analysers
H. J. NOEBELS
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I. S. PAVLUSHENKO, A. V. YANISHEVSKII
J. Appl. Chem., USSR, 1959, **32** (7), 1529-35.

The Selection and Specification of Process Equipment (Crystallizers, Dryers, Centrifuges, Filters)
D. J. ORIOLO
Ingen. quím. Méx., 1960 (45), 32-41.

Continuous Sampling and Automatic Analysis for Silica in Modern High-pressure Boilers
K. F. SCHUNK
Ann. N.Y. Acad. Sci., 1960, **87** (2), 924-33.

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D. G. STEPHAN
Mod. Castings, 1960, **38** (1), 75-83.

Production of Sodium Sulphate by the Hargreaves-Robinson Process. (Process also adapted for making K_2SO_4 from KCl and S)
E. E. ALVARADO RIOS
Ingen. quím. Méx., 1960, (47), 19-23.

* These articles do *not* include those appearing in **Abstracts**.

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